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March 11 , 1991

Ms. Marcia Bailey
U.S. Environmental Protection Agency
1200 Sixth Avenue
Seattle, WA 98101

RE: Contract No. 68-W9-0009, Work Assignment No. 112R10047, Quality Assurance Project Plan (QAPjP) for the Ridgefield Brick and Tile site, Ridgefield, Washington

Dear Ms. ~~Bailey~~: Marcia

PRC Environmental Management, Inc., (PRC) is pleased to submit two copies of the enclosed QAPjP for the Ridgefield Brick and Tile site in Ridgefield, Washington. Appendix A contains Special Analytical Services (SAS) requests for PAH, chlorophenols, and metals. The SAS requests are also provided on a floppy disk (WordPerfect 5.0) as requested by the EPA Quality Assurance Office.

If you have any questions or comments, please contact me at (206) 624-2692.

Sincerely,

PRC ENVIRONMENTAL MANAGEMENT, INC.

Gary A. Bruno

Gary A. Bruno
Environmental Geologist

Enclosures

cc: Laura Castrilli, EPA QAO, Seattle
Dave Liu, PRC, Chicago

USEPA RCRA
3057992

RIDGEFIELD BRICK
AND TILE
RIDGEFIELD, WASHINGTON

OPERATION AND MAINTENANCE INSPECTION

QUALITY ASSURANCE PROJECT PLAN

Prepared For

U.S. ENVIRONMENTAL PROTECTION AGENCY
Region 10
Seattle, Washington

Work Assignment No.	:	112R10047
EPA Region	:	10
EPA I.D. No.	:	WAD 009422411
Date Prepared	:	March 11, 1991
Contract No.	:	068-W9-0009
PRC No.	:	112-R1004707
Prepared by	:	PRC Environmental Management, Inc. Gary Bruno
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RIDGEFIELD BRICK
AND TILE
RIDGEFIELD, WASHINGTON

OPERATION AND MAINTENANCE INSPECTION

Revision No. 0

March 11, 1991

TITLE AND APPROVAL PAGE

Approvals:

______________________________ ______________
Marcia Bailey
U.S. EPA Region 10 Work Assignment Manager
Date

______________________________ ______________
Barry Towns
U.S. EPA Region 10 Quality Assurance Officer
Date

James F. Pankanin ______________ 3/11/91
James Pankanin
PRC Project Manager
Date

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1.0 PROJECT DESCRIPTION

The U.S. Environmental Protection Agency (EPA) requested PRC Environmental Management, Inc. (PRC) perform an operation and maintenance (O&M) inspection at the Ridgefield Brick and Tile (RBT) site in Ridgefield, Washington (EPA identification number WAD 009422411). The O&M is being performed as work assignment number 12R10047 under contract number 68-W9-0009 Technical Enforcement Support (TES) 12.

PRC prepared this quality assurance project plan (QAPjP) to satisfy EPA quality assurance and quality control (QA/QC) requirements for receiving and analyzing split ground-water and leachate samples at the RBT site. This QAPjP discusses data quality objectives (DQOs) and outlines QA/QC procedures for split sampling and analytical determination of hazardous waste constituents in ground-water and leachate at the RBT site.

1.1 SITE DESCRIPTION AND HISTORY

The RBT site is located at 3510 N.W. 289th Street, Ridgefield, Clark County, Washington in a rural area. The site is in the northwest quarter of the southeast quarter of Section 17, Township 4 North, Range 1 East, of the Willamette Meridian (Figure 1-1). The inactive landfill covers about 0.75 acres on the east portion of the site (Figure 1-2).

The RBT site is owned by the Pacific Wood Treating Corporation (PWT) who owns and operates an active wood treating facility in Ridgefield, Washington. PWT disposed of about 5,100 cubic yards of potentially contaminated log decking and yard cleanup waste and about 2,500 cubic yards of boiler ash at the RBT landfill between 1979 and 1982 originating from the wood treating facility. Of the total boiler ash, only an estimated 5 cubic yards of ash has been derived from the incineration of a wastewater treatment sludge designated as a K001/D004 hazardous waste.

PWT has used several wood preservatives, including pentachlorophenol, creosote, and chrome/copper/arsenic solutions at its wood treating facility in Ridgefield. PWT has burned an estimated 20 million lbs/year of waste wood at its treating facility boiler, and, from 1979 to 1982, it burned about 32,000 lbs/year of a wastewater sludge containing the treating solutions described above. Due to its treating solution content, the sludge has been designated a Resource

Conservation and Recovery Act (RCRA)-listed and -characteristic hazardous waste (K001 and D004). Ash derived from the incineration of the K001/D004 sludge retains its RCRA hazardous waste designation. Additionally, any other solid waste mixed with the K001/D004 sludge ash, such as log decking and yard cleanup waste, also becomes RCRA-listed hazardous waste.

During an EPA inspection at the PWT Ridgefield wood treating facility, EPA discovered that PWT was disposing of the RCRA-listed and -regulated K001/D004 hazardous waste at the RBT landfill. Subsequently, PWT submitted a RCRA Part A permit application for the RBT landfill on May 23, 1983, thus obtaining interim status for the disposal facility. PWT submitted a closure plan for the RBT landfill to the Washington State Department of Ecology (Ecology) and conducted closure activities in September 1983, reportedly under Ecology supervision. As a result of deficiencies in the original closure plan and closure activities, EPA issued a Consent Agreement and Final Order to PWT in November 1986. As required by the order, PWT submitted a revised closure plan in February 1987 which was also determined by EPA to be deficient. An updated closure plan is still pending, however, a ground-water monitoring system was installed at the RBT site in August 1988.

PWT has monitored on-site lysimeters, monitoring wells, and leachate from the landfill toe drain on several occasions since 1983. Concentrations of pentachlorophenol and naphthalene in both ground water and leachate have been typically below 2 micrograms per liter (μg/L) and always below 10 μg/L. Concentrations of arsenic and chromium in ground water and leachate have been typically below drinking water standards of 50 μg/L.

1.2 O&M PURPOSE

PRC is conducting an O&M at the RBT site based on the objectives of the final O&M inspection guidance document for RCRA ground-water monitoring systems (EPA, 1988d) and the EPA O&M statement of work. Generally, the O&M will evaluate how the facility operates and maintains its ground-water monitoring system in compliance with RCRA regulations, permit requirements, or other order requirements to which it is subject.

1.3 O&M OBJECTIVES

The specific RBT O&M objectives are to:

- Evaluate if the facility's ground-water monitoring system is in compliance with RCRA interim status ground-water monitoring regulations (40 CFR 265, Subpart F) and the Consent Agreement and Final Order issued to RBT in November 1986 (EPA, 1986d).

- Evaluate the facility's sampling protocol and methods including adherence to its approved or current sampling and analysis plan. The plan will also be evaluated on its technical merits.

- Evaluate the adequacy of the facility's analytical program and performance by receiving and analyzing split ground-water and leachate samples.

- Evaluate the facility's maintenance of its existing ground-water monitoring system by determining that field sampling devices are in working order, that the facility is abiding by maintenance provisions outlined in its sampling and analysis plan, and that individual monitoring wells in the ground-water monitoring system yield representative ground-water samples.

Additionally, EPA is considering a clean closure option for the RBT landfill. For this alternative, EPA needs supporting analytical data to aid in site characterization. Therefore, EPA has requested that analyses be performed on the split ground-water and leachate samples for certain parameters (including polynuclear aromatic hydrocarbons (PAH), chlorophenols, arsenic, and chromium) using EPA-approved analytical methods having detection limits at or below 1.0 μg/L. Split samples will also be received and analyzed for volatile organic compounds (VOCs) using method detection limit criteria as specified in the Contract Laboratory Program Statement of Work for Organic Analysis (EPA, 1990a). Summary information regarding the RBT sampling program is provided in Tables 3-1, 4-1, and 4-2.

1.4 O&M INSPECTION ACTIVITIES

PRC will meet the objectives and additional data needs described above by observing RBT field sampling activities and receiving split ground-water and leachate samples. PRC will evaluate whether the RBT sampling activities follow the requirements in 40 CFR 265 Subpart F, the Consent Agreement and Final Order, the RBT sampling and analysis plan, and generally accepted ground-water sampling procedures specified in the RCRA ground-water monitoring technical enforcement guidance (U.S. EPA, 1986d). PRC will monitor the following specific RBT sampling activities during the sampling event:

- Depth-to-water measurements
- Field measurements of water quality parameters (pH, specific conductance, and temperature)
- Well purging
- Sample collection
- Equipment decontamination
- Quality assurance procedures
- Chain-of-custody procedures

PRC will document any inconsistencies or deficiencies in RBT sampling procedures in an O&M report.

In addition to performing observation activities, PRC will also receive split ground-water and leachate samples from on-site monitoring wells and the landfill toe drain system. The split-samples will be analyzed by the EPA Region 10 Manchester laboratory or a Contract Laboratory Program (CLP) laboratory, depending on availability, for site-specific parameters including VOCs, PAH, chlorophenols, arsenic, and chromium using the methods specified in the QAPjP. Summary information regarding the RBT sampling program is provided in Tables 3-1, 4-1, and 4-2.

1.5 SCHEDULE OF PROJECT ACTIVITIES

The anticipated schedule of O&M events and deliverables is as follows:

Activity	Dates
Sampling and Analysis	
Field split sampling	March 27 and 28, 1991
Sample analysis (by EPA Region 10 Manchester laboratory or CLP laboratory)	Week of March 25, 1991
Deliverables	
EPA approved QAPjP	March 13, 1991
EPA approved Health and Safety Plan	March 13, 1991
PRC data validation report	30 days after receipt of CLP data (approximately June 21, 1991)
O&M report	60 days after receipt of EPA, CLP, and facility data (approximately July 21, 1991)

1.6 DATA USAGE

The O&M conducted at RBT will generate two types of data:

- Inspection information
- Analytical data for split ground-water and leachate samples

PRC will use the inspection information to evaluate how the facility operates and maintains its ground-water monitoring system. PRC will use the split-sample analytical data to specifically evaluate the adequacy of the facility's analytical program and performance. The split-sample analytical data will also be used by EPA to aid in characterizing the site in support of its clean closure option proposed by EPA for the RBT landfill.

1.7 SAMPLING RATIONALE

1.7.1 Monitoring Well System

PRC will receive split ground-water samples from the RBT site. The ground-water monitoring system consists of seven monitoring wells placed around the inactive landfill. The wells are screened in the upper stratigraphic units of alluvial sands, silts, and clays (20 to 25 feet thick) and a lower unit of weathered gravel. Wells B-2 and B-5 are upgradient and wells B-1, B-3, B-4, B-6, and B-7 are downgradient wells based on the flow direction of an inferred perched ground-water aquifer beneath the RBT site (PWT, 1988).

The seven monitoring wells, installed in August 1987, monitor both a sand interbed unit and underlying gravels. During wet periods, a perched ground-water condition exists in the sand and gravel units. Monitoring wells B-1, B-5, B-6, and B-7 are constructed with screened sections in the sand interbed and underlying gravels. Monitoring wells B-2, B-3, and B-4 are constructed in areas where the sand interbed is not encountered, and therefore, are screened in the top portions of the underlying gravels. Additionally, the screened portions of monitoring wells B-2 and B-3 extend upward into the shallow clayey silt unit (PWT, 1988). Refer to Figure 1-3 for monitoring well details.

RBT field sampling from 1987 to the present has shown that ground water in the monitoring well system is temporary and seasonal. Monitoring wells B-1, B-5, and B-6 have had 8 inches to 9 feet of ground water. Monitoring wells B-2, B-3, and B-4 have only had small amounts of ground water. Monitoring well B-7 has been dry (PWT, 1988). Apparently, the main aquifer underlying the RBT site is 180-220 feet deep, located in sands and gravels of the underlying lower Troutdale Formation. The ground-water flow direction in the main aquifer is believed to be to the northwest. However, a detailed evaluation of the flow characteristics beneath the RBT site has not been made (Tetra Tech, 1989).

PRC has selected the following monitoring wells for split sampling at the RBT site in order to receive representative ground-water samples from upgradient and downgradient locations, various geologic units (sand interbed, gravel, and clay units), as well as provide the greatest chance that ground water will be available to sample during the scheduled March 1991 sampling event. Wells in the monitoring well system go dry as summer approaches. Well selection is summarized as follows:

Monitoring Well	Criteria
B-5	Upgradient; Screened in sand interbed unit
B-3	Downgradient; Screened in gravel/clay unit
B-4	Downgradient; Screened in gravel unit
B-6	Downgradient; Screened in sand interbed unit

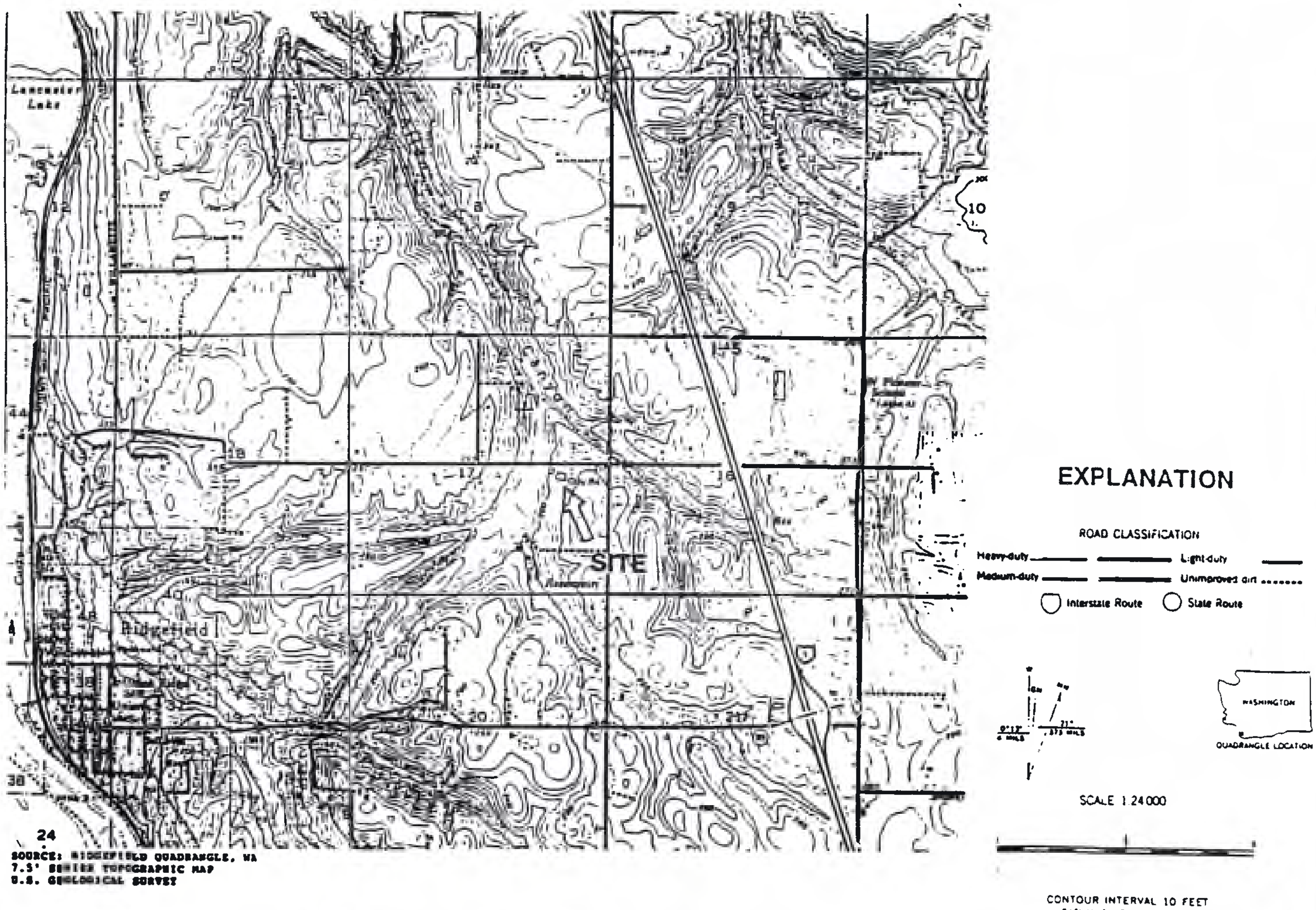
Since monitoring well B-6 has had measurable levels of ground water and is downgradient of the landfill, it will be designated for receiving of additional quality assurance/quality control (QA/QC) samples, including a duplicate sample and matrix spike/matrix spike duplicate (MS/MSD) samples (refer to Section 3.4 and 8.0).

1.7.2 Leachate Collection System

PRC will also receive split samples of leachate from the RBT landfill leachate collection system (toe drain). Since January 1986, PWT has contracted a disposal company to empty the leachate collection tank about every nine months. About 900 gallons of leachate has reportedly been disposed of each time. The toe drain is located at the west edge of the landfill. The toe drain consists of an 8-foot deep vertical section of steel pipe, about 3 feet in diameter (Tetra Tech, 1989).

PRC will receive representative samples from the leachate collection system by first split sampling the standing leachate in the toe drain. PRC will receive an environmental sample, a duplicate sample, and MS/MSD samples from the standing leachate to be assured of an adequate volume of leachate for all samples. PWT will then purge the toe drain. PRC will then receive a split environmental sample from the inlet pipe (draining the landfill) near the bottom of the toe drain.

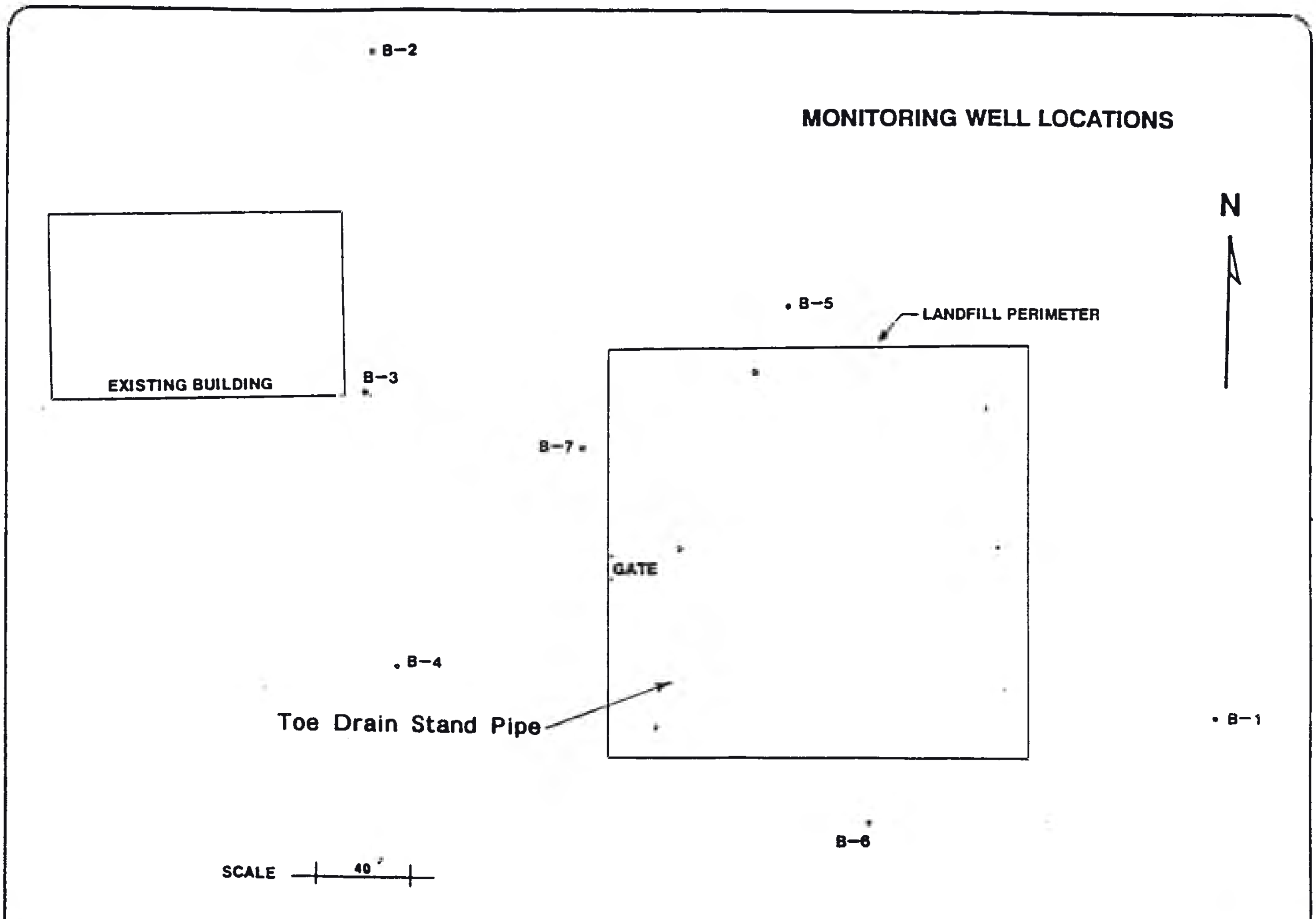
FIGURE 1-1
SITE LOCATOR MAP



Source: Adapted from PWT, 1988

FIGURE 1-2

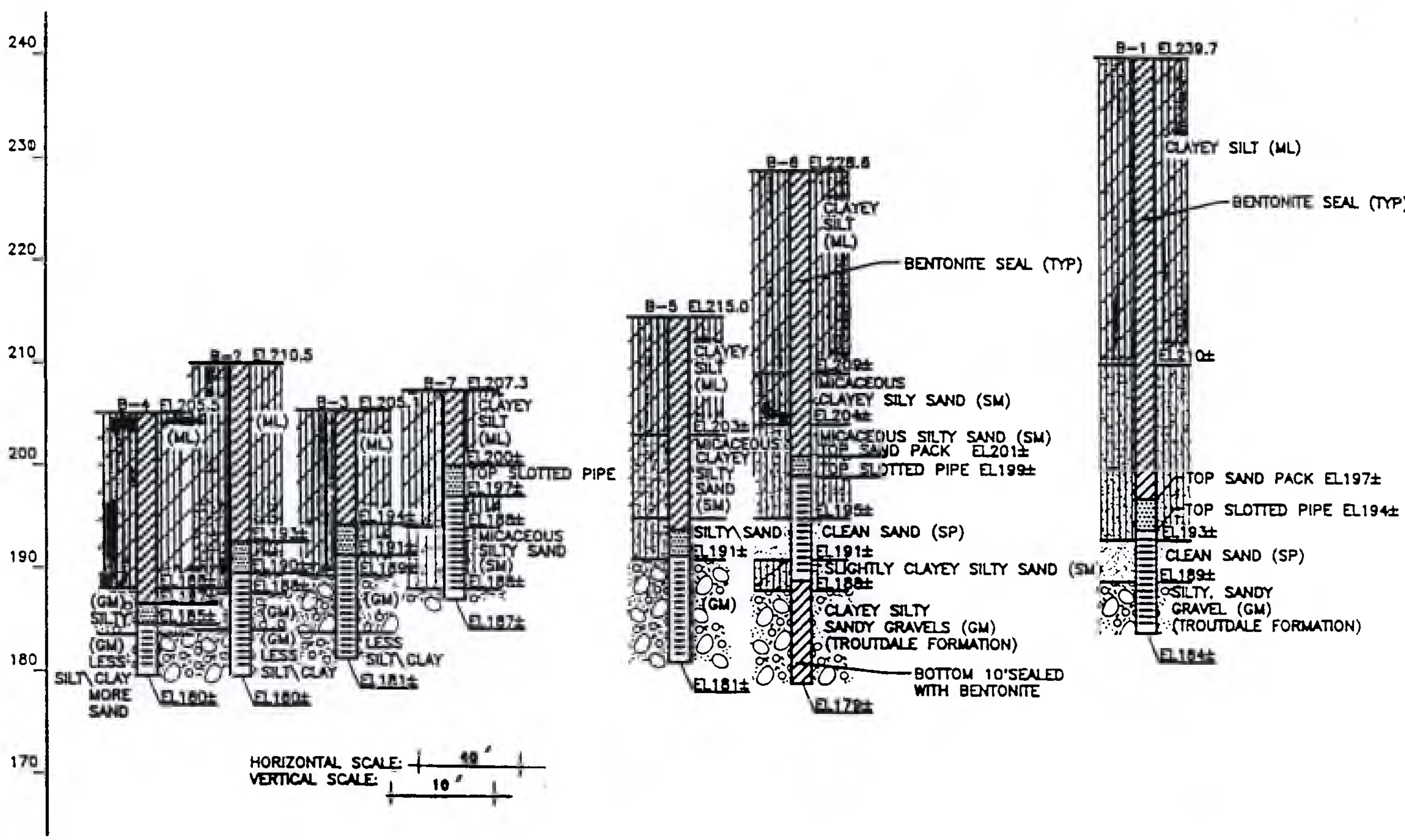
DETAILED SITE MAP



Source: Adapted from PWT, 1988

FIGURE 1-3

MONITORING WELL CONSTRUCTION DIAGRAMS



Source: Adapted from PWT, 1988

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The EPA work assignment manager (WAM) has primary responsibility for the RBT O&M. PRC is responsible for conducting field sampling activities, validating data, and reporting results. A project organization chart outlining major QA/QC responsibilities for this project is presented in Figure 2-1.

The rest of this section outlines responsibilities and responsible individuals for four separate aspects of the O&M: management, QA/QC, field operations, and laboratory services.

2.1 MANAGEMENT RESPONSIBILITIES

Responsibility for execution and management of technical and administrative aspects of the RBT O&M has been assigned as follows:

- EPA Regional Project Officer (RPO) (Vicky Tapang) -- Overall management of TES 12 RCRA work assignments
- EPA WAM (Marcia Bailey) -- Management of the RBT O&M
- PRC Regional Manager (Jim Pankanin) -- Overall management of all PRC TES 12 work assignments in EPA Region 10
- PRC Project Manager (Jim Pankanin) -- Management of the RBT O&M for PRC

2.2 QUALITY ASSURANCE RESPONSIBILITIES

The following organizations and individuals are responsible for QA/QC of the O&M conducted under this QAPjP:

- EPA WAM (Marcia Bailey) -- Review and approval of QAPjP; review and approval of O&M report
- EPA Region 10 QA Officer (Barry Towns) -- Review and approval of QAPjP
- PRC TES 12 QA Manager (Dave Liu) -- Overall QA for TES 12 work assignments
- PRC Regional QAPjP Technical Monitor (Jeff Ross) -- Technical review of QAPjP
- PRC Project Manager (Jim Pankanin) -- Approval of QAPjP

2.3 FIELD SAMPLING RESPONSIBILITIES

PRC will be responsible for performing all field sampling activities specified in this QAPjP under the direction of EPA. Specific field sampling responsibilities are as follows:

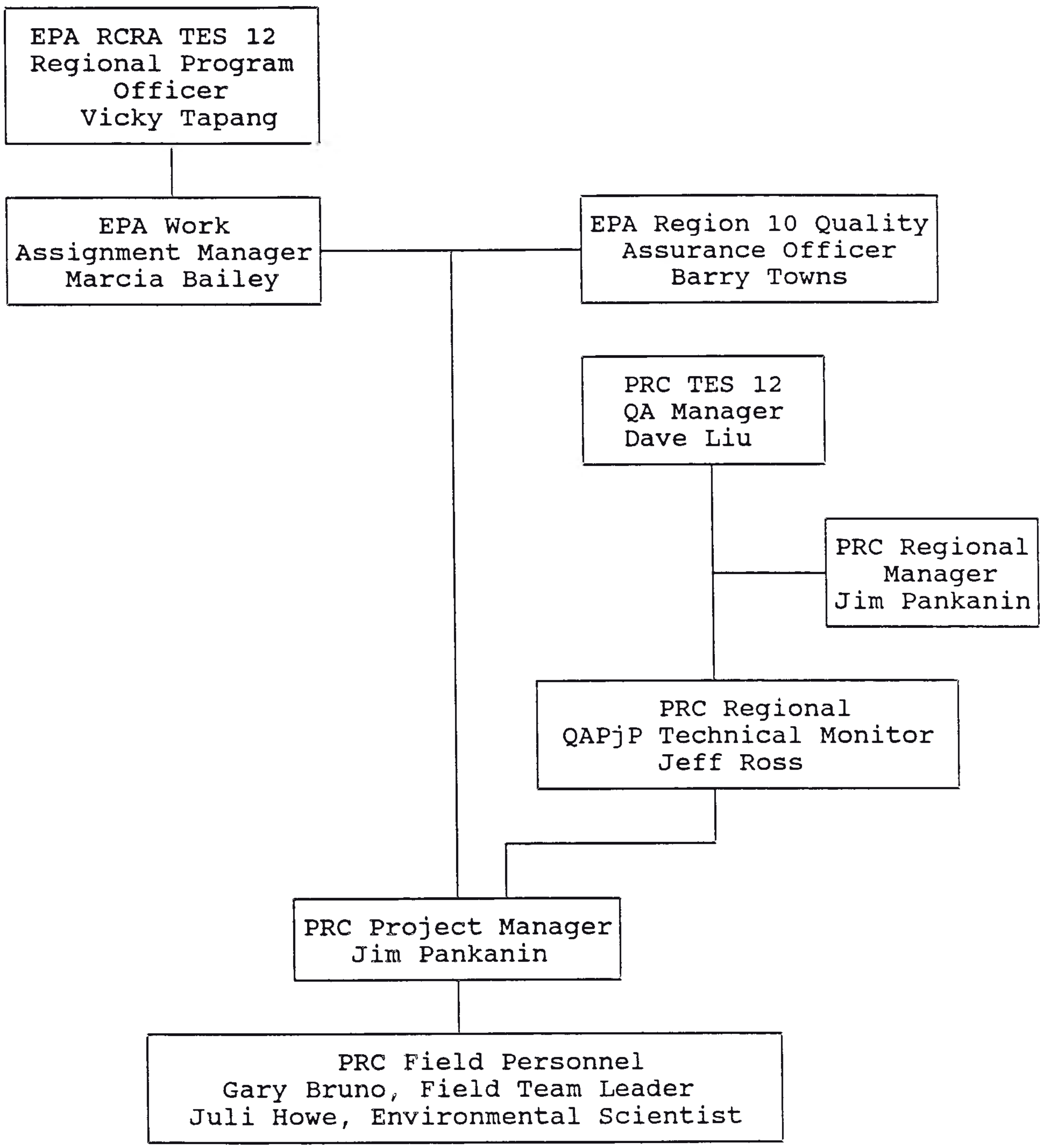
- EPA WAM (Marcia Bailey) -- Overall direction of field sampling
- PRC Project Manager (Jim Pankanin) -- Technical direction of field sampling
- PRC Field Team Leader (Gary Bruno) -- Direction and coordination of field sampling in accordance with this QAPjP

2.4 LABORATORY RESPONSIBILITIES

Laboratory analysis for split ground-water and leachate samples received during the O&M will be conducted through the EPA Region 10 Manchester Laboratory or a CLP laboratory, depending on laboratory availability. The EPA QA Officer (Barry Towns) will coordinate and manage all EPA and CLP responsibilities for split ground-water and leachate samples received during the O&M.

FIGURE 2-1

PROJECT ORGANIZATION CHART



3.0 QUALITY ASSURANCE/QUALITY CONTROL OBJECTIVES

The purpose of this section is to address QA/QC objectives for completeness, representativeness, comparability, precision, and accuracy of data. The overall objective is to develop and implement procedures for field sampling activities, chain-of-custody, laboratory analysis, and reporting that will promote high quality data from the O&M. Sections 3.1, 3.2, and 3.3 discuss QA/QC objectives for completeness, representativeness, and comparability. Section 3.4 discusses QA/QC objectives for precision and accuracy for CLP Routine Analytical Services (RAS), CLP Special Analytical Services (SAS), and field measurements.

3.1 COMPLETENESS

Completeness is measured by the amount of valid analytical data obtained compared to the amount of analytical data expected under normal conditions. For this QAPjP, PRC has defined "the amount of analytical data expected under normal conditions" as the total number of environmental samples planned to be received and analyzed for each system. That is, four samples are planned to be received and analyzed for the ground-water monitoring system and two samples for the leachate collection system. Based on six samples planned to be received and analyzed for this O&M, the completeness criteria for both the field and laboratory will be 80 percent.

3.2 REPRESENTATIVENESS

Representativeness is the degree to which sample data represent a characteristic of a population, parameter variation at a sampling point, or an environmental condition. The sampling locations for this O&M were selected to receive ground-water and leachate samples that will adequately determine if there is a release to the environment at the RBT site (Section 1.7).

Representativeness is enhanced when all samples from a particular medium are collected (received) using the same technique. For this effort, PRC will receive ground-water and leachate samples according to sampling procedures outlined in Section 4.0

Representativeness is also achieved by assuring that sampling equipment is properly decontaminated between sampling locations. Field equipment for the ground-water and leachate sampling will be decontaminated between samples to avoid cross contamination of samples collected (received) at subsequent locations (Section 4.3). RBT will be responsible for decontaminating field sampling equipment.

3.3 COMPARABILITY

Comparability expresses the confidence with which one data set can be compared to another. To assure that ground-water and leachate sample results are comparable to future sample results, PRC will document all sample locations, conditions, field sampling methods, and laboratory analysis methods.

3.4 PRECISION AND ACCURACY

Precision and accuracy are indicators of data quality. Generally, precision is a measure of the variability of a group of measurements compared to their mean value. Sampling and analytical precision is determined by analyzing field duplicate samples. Accuracy is a measure of the bias in a measurement system. Sampling accuracy is assessed by analyzing equipment rinsate field blanks, trip blanks, and field (transfer) blanks, while analytical accuracy is assessed by analyzing surrogate and matrix spike samples. The type of QA/QC samples to be received for determining precision and accuracy are described below as well as Section 8.0. Precision and accuracy objectives for CLP RAS, CLP SAS, and field sample analysis are also described below and summarized in Table 3-1.

3.4.1 Types of Quality Assurance/Quality Control Samples

PRC will receive five types of QA/QC samples to determine precision and accuracy: field duplicates, equipment rinsate field blanks, trip blanks, field (transfer) blanks, and MS/MSD samples. One duplicate sample will be received from each system (monitoring well and leachate collection) and submitted for laboratory analysis to determine sampling and analytical precision. Equipment rinsate field blanks will also be received at each system and submitted for laboratory analysis to check for contamination potentially occurring from sampling equipment used at the site (that is, the thoroughness of decontamination procedures) (sampling accuracy).

One trip blank sample will be included in every cooler shipped to the laboratory that contains environmental samples for VOC analysis. The trip blanks will be analyzed for target compound list (TCL) VOCs to check for contamination potentially occurring during shipping and handling (sampling accuracy).

One field (transfer) blank will be prepared per day of sampling. The field (transfer) blank will be analyzed for TCL VOCs to check for potential contamination occurring from ambient conditions (sampling accuracy).

MS/MSD samples will also be received from each system. MS/MSD samples will be analyzed and used to determine analytical accuracy. Percent recovery values for these samples will be compared to acceptance criteria in the CLP SOWs (Section 12.0).

3.4.2 CLP Routine Analytical Services (RAS) Quality Assurance/Quality Control Objectives

For CLP RAS parameters, the criteria for precision and accuracy are defined by the CLP Statements of Work (SOWs) for organic analysis (EPA, 1990a) and inorganic analysis (EPA, 1990b) and will serve as DQOs. Desired method detection limits for this O&M will follow CLP RAS parameters. Table 3-1 summarizes the QA/QC objectives for the CLP RAS parameters.

3.4.3 CLP Special Analytical Services (SAS) Quality Assurance/Quality Control Objectives

CLP SAS analyses will be required to analyze ground-water and leachate samples for PAH, chlorophenols, arsenic, and chromium. An equipment rinsate field blank, a field duplicate, and MS/MSD samples will be received and analyzed at the same frequency as for CLP RAS parameters. The SAS request form in Appendix A includes precision and accuracy criteria for the PAH, chlorophenol, arsenic, and chromium analyses. Table 3-1 also summarizes the QA/QC objectives for the CLP SAS parameters.

3.4.4 Field Quality Assurance/Quality Control Objectives

Field measurements for volatile organic vapors will be made at the RBT site for health and safety reasons using an HNU model P-101 photoionization detector. Field measurement, calibration, and maintenance procedures for the HNu are described in the operator's manual in Appendix B.

TABLE 3-1
DATA QUALITY OBJECTIVES FOR THE RBT O&M

Parameters	Method Detection Limit (µg/L)	Precision (Relative Percent Difference)	Accuracy (Percent Spike Recovery)	Completeness	Analytical Methods
VOC	Per Method	±20	75 - 125	80	CLP RAS (a)
PAH	0.013 - 2.3 (b)	±20	70 - 130	80	CLP SAS (c) -SW-846 Method (d) (3520/3620/8310)
Chlorophenols	1.0	±20	70 - 130	80	CLP SAS (c) -SW-846 Method (d) (Modified 8040)
Total Arsenic & Chromium	1.0	±20	90 - 110	80	CLP SAS (c) EPA Method (e) (218.2 Chromium) (206.2 Arsenic)
Dissolved Arsenic & Chromium	1.0	±20	90 - 110	80	CLP SAS (c) EPA Method (e) (218.2 Chromium) (206.2 Arsenic)

a EPA Contract Laboratory Program Routine Analytical Services (EPA, 1990a)

b See Method 8310 (EPA, 1986a) for detection limits for specific compounds.

c EPA Contract Laboratory Program Special Analytical Services (see Appendix A)

d EPA 1986a

e EPA 1983

4.0 SAMPLING PROCEDURES

PRC will perform split ground-water and leachate sampling at the RBT site as part of the O&M. PRC will also receive and submit appropriate QA/QC samples for each system (monitoring well and leachate collection). The QA/QC samples are discussed in Sections 3.0 and 8.0. A summary of the RBT sampling program is presented in Table 4-1.

4.1 SPLIT GROUND-WATER SAMPLING -- MONITORING WELL SYSTEM

PRC will receive split ground-water samples from four on-site monitoring wells (B-3, B-4, B-5, B-6) (see Table 4-1 and Figure 1-2). As discussed in Section 1.7.1, these four monitoring wells represent both upgradient and downgradient locations and various geologic units at the site (sand interbed, gravel, and clay). These wells also have the greatest chance of having ground water available for split sampling during the March 1991 sampling event. Wells in the monitoring well system go dry as summer approaches. Well selection is summarized as follows:

Monitoring Well	Criteria
B-5	Upgradient; Screened in sand interbed unit
B-3	Downgradient; Screened in gravel/clay unit
B-4	Downgradient; Screened in gravel unit
B-6	Downgradient; Screened in sand interbed unit

PRC will also receive QA/QC samples including a duplicate sample and MS/MSD samples (at well B-6), an equipment rinsate field blank, a trip blank and a field (transfer) blank (refer to Table 4-1). QA/QC samples are discussed in detail in Section 8.0.

To receive split ground-water samples, PRC will provide the appropriate sample containers, preservatives, shipping coolers, and miscellaneous field supplies (see Table 4-2). PRC will receive split samples for each parameter immediately after PWT collects its ground-water samples for that parameter. For each sample parameter, PWT will fill PRC's containers for that parameter with ground water from the same bailer (when possible) used by PWT to collect its similar sample parameter. When the volume of ground water required for a sample necessitates using more than one sample container, PWT's and PRC's containers for that sample will be filled alternately (one container at a time) until all containers are filled.

PRC will follow the PWT field filtering protocol for filtered (dissolved) metals samples. That is, similar to receiving other split samples, PRC will provide the appropriate containers and receive the filtered metal samples from PWT. If PWT does not collect field filtered metals samples as part of it sampling effort, PRC will provide field filtering equipment (0.45 micron-filtering containers and hand-held vacuum pump). In this case, PRC will collect the filtered metals samples by holding its field filtering containers to be filled by PWT. PRC will filter the samples using the hand-held vacuum pump, then transfer the filtered samples to the appropriate type and number of containers. After each split ground-water sample set is received by PRC at each well (or potentially collected by PRC in the case of filtered metals samples), PRC will preserve the PAH, chlorophenol, and total and dissolved metals samples. VOC samples will be preserved prior to filling the VOA vials. VOA vials will also be filled with no headspace. Table 4-2 specifies the holding times, preservation, and containers required for the RBT sampling event.

PRC's split ground-water samples and QA/QC samples will be analyzed by the EPA Region 10 Manchester laboratory or a CLP laboratory, depending on availability. The samples will be analyzed as a CLP RAS request for VOC and CLP SAS requests for PAH, chlorophenols, arsenic, and chromium (refer to Table 3-1, Section 3.0).

4.2 SPLIT LEACHATE SAMPLING -- TOE DRAIN

PRC will also receive split leachate samples from the landfill leachate collection system (toe drain) (see Table 4-1 and Figure 1-2). As discussed in Section 1.7.2, PRC will receive representative samples by first receiving one split environmental sample, a duplicate sample, and MS/MSD samples from the standing leachate in the toe drain to be assured of an adequate volume of leachate for all samples. PWT will then purge the toe drain. PRC will then receive a split environmental sample from the inlet pipe (draining the landfill) near the bottom of the toe drain (refer to Table 4-1). The collection (receiving) and preparation of QA/QC samples are discussed in detail in Section 8.0.

The PRC split-sampling protocol for receiving leachate samples is similar to that discussed for receiving ground-water samples (Section 4.1). The leachate samples will be analyzed as a CLP RAS request for VOCs and CLP SAS requests for PAH, chlorophenols, arsenic, and chromium (refer to Table 3-1, Section 3.0).

PRC personnel will document all field activity in a bound logbook as described in Section 5.1.2. PRC will use EPA and CLP sample numbers issued by the EPA Regional Sample Control Center (RSCC).

4.3 DECONTAMINATION PROCEDURES

Decontamination of sampling equipment used at the RBT site will be performed by RBT following its sampling and analysis plan. The receiving of equipment rinsate field blanks are described in Section 8.1.2. PRC does not anticipate any contamination of nondisposable items during the receiving of split samples. Contaminated disposable items such as latex gloves, ground-water filters, and used (empty) collection containers will be placed in plastic garbage bags and disposed of by RBT.

TABLE 4-1
SUMMARY OF SAMPLING PROGRAM FOR THE RBT O&M

Sample Location[a] (System)	Sample Matrix	Parameters[b]	Environmental Samples[c]	Environmental Field Duplicate[d]	Rinsate Field Blank[e]	Trip Blank[f]	Field (Transfer) Blank[g]
1) Groundwater Monitoring Well System							
B-3	Water (split grab sample)	VOC	1				
		PAH	1				
		Chlorophenols	1				
		Total Arsenic & Chromium	1				
		Dissolved Arsenic & Chromium	1				
B-4	Water (split grab sample)	VOC	1				
		PAH	1				
		Chlorophenols	1				
		Total Arsenic & Chromium	1				
		Dissolved Arsenic & Chromium	1				
B-5	Water (split grab sample)	VOC	1				
		PAH	1				
		Chlorophenols	1				
		Total Arsenic & Chromium	1				
		Dissolved Arsenic & Chromium	1				
B-6 (MS/MSD)[h]	Water (split grab sample)	VOC	1				
		PAH	1				
		Chlorophenols	1				
		Total Arsenic & Chromium	1				
		Dissolved Arsenic & Chromium	1				

TABLE 4-1 (Continued)
SUMMARY OF SAMPLING PROGRAM FOR THE RBT O&M

Sample Location[a] (System)	Sample Matrix	Parameters[b]	Environmental Samples[c]	Environmental Field Duplicate[d]	Rinsate Field Blank[e]	Trip Blank[f]	Field (Transfer) Blank[g]
B-10 (duplicate of B-6)	Water (split grab sample)	VOC		1			
		PAH		1			
		Chlorophenols		1			
		Total Arsenic & Chromium		1			
		Dissolved Arsenic & Chromium		1			
B-15	Water (blank)	VOC			1		
		PAH			1		
		Chlorophenols			1		
		Total Arsenic & Chromium			1		
		Dissolved Arsenic & Chromium			1		
B-20	Water (blank)	VOC				2	
B-25	Water (blank)	VOC					1
TOTAL SAMPLES (System No. 1)		VOC	4	1	1	2	1
		PAH	4	1	1		
		Chlorophenols	4	1	1		
		Total Arsenic & Chromium	4	1	1		
		Dissolved Arsenic & Chromium	4	1	1		

TABLE 4-1 (Continued)
SUMMARY OF SAMPLING PROGRAM FOR THE RBT O&M

Sample Location[a] (System)	Sample Matrix	Parameters[b]	Environmental Samples[c]	Environmental Field Duplicate[d]	Rinsate Field Blank[e]	Trip Blank[f]	Field (Transfer) Blank[g]
2) Leachate Collection System (Toe Drain)							
TD-1 (MS/MSD)[i]	Water (split grab sample - standing leachate)	VOC	1				
		PAH	1				
		Chlorophenols	1				
		Total Arsenic & Chromium	1				
		Dissolved Arsenic & Chromium	1				
TD-2	Water (split grab sample - inlet leachate)	VOC	1				
		PAH	1				
		Chlorophenols	1				
		Total Arsenic & Chromium	1				
		Dissolved Arsenic & Chromium	1				
TD-10 (duplicate of TD-1)	Water (split grab sample - standing water)	VOC		1			
		PAH		1			
		Chlorophenols		1			
		Total Arsenic & Chromium		1			
		Dissolved Arsenic & Chromium		1			
TD-15	Water (blank)	VOC			1		
		PAH			1		
		Chlorophenols			1		
		Total Arsenic & Chromium			1		
		Dissolved Arsenic & Chromium			1		

TABLE 4-1 (Continued)
SUMMARY OF SAMPLING PROGRAM FOR THE RBT O&M

Sample Location[a] (System)	Sample Matrix	Parameters[b]	Environmental Samples[c]	Environmental Field Duplicate[d]	Rinsate Field Blank[e]	Trip Blank[f]	Field (Transfer) Blank[g]
TOTAL SAMPLES (System No. 2)		VOC	2	1	1		
		PAH	2	1	1		
		Chlorophenols	2	1	1		
		Total Arsenic & Chromium	2	1	1		
		Dissolved Arsenic & Chromium	2	1	1		

Notes:

a B = Monitoring well designation
TD = Toe Drain

b See Table 3-1

c Matrix spike/Matrix spike duplicate (MS/MSD) samples are required for CLP RAS and SAS analyses. One set of MS/MSD samples each will be collected at B-6 (monitoring well system) and at TD-2 (leachate collection system). Triple volumes are required for VOC, PAH, and chlorophenols (organic) analyses; Double volumes are required for arsenic and chromium (inorganic) analyses. However, MS/MSD samples do not count toward the sample total and are not included in the table as a separate item.

d One field duplicate will be taken at B-6 and TD-2 or one for every 10 total samples.

e Equipment rinsate field blanks will be prepared in the field by pouring carbon-free water over decontaminated ground-water sampling equipment.

f Each trip blank will consist of two 40-ml VOA vials filled with carbon-free water by the analytical laboratory. The trip blanks will be shipped with the other samples for VOC analyses. One trip blank will be shipped with each cooler containing VOC samples.

g The field (transfer) blank will consist of two 40-ml VOA vials filled with carbon-free water in the field by PRC personnel

TABLE 4-2
SAMPLE HOLDING TIME, PRESERVATION, AND CONTAINER REQUIREMENTS

Parameters	Matrix	Holding Times[a]	Preservation[a]	Containers per Sample, Blank, or MS/MSD[a]	Total Samples and Blanks	Total Containers Required (Including MS/MSD)[b]
VOC	Water	14 days	Four drops concentrated HCl. Cool to 4°C	2 x 40-ml glass vials, Teflon-lined septum caps	13	34
PAH	Water	7 days until extraction, 40 days after extraction	1.5 ml 10% $Na_2S_2O_3$ per L; Store in dark; Cool to 4°C	4 x 1-L amber glass, Teflon-lined septum caps	10	56
Chlorophenols	Water	7 days until extraction, 40 days after extraction	1.5 ml 10% $Na_2S_2O_3$ per L; Cool to 4°C	4 x 1-L amber glass, Teflon-lined septum caps	10	56
Total Arsenic & Chromium	Water	6 months	HNO_3 to pH<2	1 x 1-L polyethylene	10	12
Dissolved Arsenic & Chromium	Water	6 months	Field filter using 0.45-micron screen; HNO_3 to pH<2	1 x 1-L polyethylene	10	12

[a] EPA, 1986a

[b] Matrix spike/Matrix spike duplicate (MS/MSD) samples are required for CLP RAS and SAS analyses. One set of MS/MSD samples each will be collected at B-6 (monitoring well system) and at TD-2 (leachate collection system). Triple volumes required for VOC, PAH, and chlorophenol (organic) analyses; Double volumes required for arsenic and chromium (inorganic) analyses.

5.0 SAMPLE DOCUMENTATION AND CUSTODY

The possession and handling of each sample processed will be properly documented to promote timely, correct, and complete analysis for all parameters requested. Most importantly, each sample must be traceable from the point of collection (receiving) through analysis and final disposition to promote sample integrity. This integrity precludes any possibility of the analytical data or subsequent conclusions being challenged in litigation or enforcement actions.

PRC will use the CLP and EPA documentation system to identify, track, and monitor each sample. This system is briefly discussed in the following sections. The EPA User's Guide to the Contract Laboratory Program (EPA, 1988a) contains further information concerning these procedures. Additional field records and control measures will be maintained according to National Enforcement Investigations Center Policies and Procedures (EPA, 1986a). Whenever questions arise, the EPA RSCC will be consulted for direction and clarification.

5.1 FIELD DOCUMENTATION AND CONTROL MEASURES

The field records, CLP, and EPA documentation control measures to be used during sample receiving, identification, handling, and shipping include the following:

- Sample tags, as shown in Figure 5-1
- Custody seals, as shown in Figure 5-1
- CLP sample analysis request forms (traffic report forms), as shown in Figures 5-2 through 5-4
- EPA Region 10 laboratory analyses request forms (organics and metals), as shown in Figures 5-5 and 5-6
- Chain-of-custody record form, as shown in Figure 5-7

All necessary CLP and EPA documentation forms, labels, seals, and other paperwork will be obtained from the EPA RSCC. The PRC project manager will be responsible for obtaining these items and distributing them to field personnel. All paperwork will be completed using indelible ink.

5.1.1 Sample Labeling

PRC will use the official EPA and CLP sample numbers issued by the EPA (RSCC) for this split sampling event. PRC will also record and cross-reference, in a bound logbook, the official EPA and CLP sample numbers with corresponding PRC split-sample designations (see Section 5.1.2). PRC's split-sample numbering system will consist of:

- A two-letter site description (RB for Ridgefield Brick)
- A multi-character sample designator (for example, monitoring well B3)
- A two-character round number (for example, 01 designating the sample frequency at the site)

For example, the split ground-water sample from monitoring well B-6 at RBT, taken on the first sampling round at the site, would be designated RB-B6-01.

Sample tags and labels will be attached to each sample container to provide proper identification of samples. The tags will be retained by the laboratory as evidence of sample receipt and analysis.

Figure 5-1 presents an example of a sample tag. The information recorded on tags and labels includes the following:

- Project code--the number assigned by EPA to the sampling project
- Lab sample number--assigned by EPA RSCC
- CLP case/RAS or SAS number(s)--the unique number(s) for CLP analyses assigned by EPA RSCC to identify the sampling event (entered under "Remarks" heading)
- CLP sample number--the unique CLP sample identification number assigned by EPA RSCC used to document the sample (entered under "Remarks" heading)
- Station location--the sampling station description as specified in the program plan
- Station number--a two-digit number assigned by the sampling team coordinator
- Date--a six-digit number indicating the month, day, and year of receiving
- Time--a four-digit number indicating the military time of receiving

- Sample type--grab or composite
- Total number of sample containers
- Samplers--signatures of sample receivers
- Remarks--Case/SAS and sample numbers, as well as any pertinent comments
- Label or tag number--a unique serial number preassigned and stamped on the label or tag

The tags and labels also have appropriate spaces for describing sample preservatives and indicating the analytical parameters to be analyzed. The completed sample tag and label will be securely attached to the sample container.

PRC will consult the EPA RSCC personnel for assistance regarding the analytical services to be used. PRC will use the appropriate analysis requests or records according to guidelines specified in the most recent CLP User's Guide (EPA, 1988a).

5.1.2 Field Logbooks

Daily field activities will be documented through journal entries in a bound logbook, dedicated to the site. The logbook will be water resistant, and all entries will be made in indelible ink. The logbook will contain all pertinent information about sampling activities, site conditions, field methodologies used, general observations, and other pertinent technical information. Examples of typical logbook entries include the following:

- Daily temperature and other climatic conditions
- Field measurements, activities, or observations
- Referenced sample location description (in relation to a stationary landmark)
- Media being sampled
- Collection (receiving) methods and equipment, including decontamination measures
- Date and time of receiving
- Types of sample containers used

- Sample identification and cross-referencing
- Sample types and preservatives used
- Parameters required for analysis
- Sample receivers, distribution, and transporters
- Site sketches
- Instrument calibration procedures and frequency

The PRC field team leader or designee will be responsible for the daily maintenance of all field data records. Each page of the logbook will be numbered, dated, and signed by the person making the entry. Corrections to the logbook will be made by using a single strike mark through the entry to be corrected and then recording and initialing the correct entry. For corrections made at a later date, the date of the correction will also be noted.

Color photographs will be taken during the O&M to document sampling locations, monitoring well maintenance, sampling activities, and other site features as necessary. The photographs will be numbered to correspond to logbook entries. The name of the photographer, date, time, site location, and photo description will be sequentially entered as the photos are taken. Adequate logbook notations and receipts will be retained to account for custody during film processing.

5.1.3 Chain-of-Custody Records

Chain-of-custody records, shown in Figure 5-7, establish the documentation necessary to trace sample possession from time of receiving through sample analysis and disposition. A sample is considered to be in an individual's custody if any of the following criteria are met:

- The sample is in a person's physical possession.
- The sample is in a person's view after being in his or her physical possession.
- The sample was in a person's physical possession and was then locked up or sealed to prevent tampering.
- The sample is kept in a secured area.

The sample receiver will complete a chain-of-custody record to accompany each sample delivery container (cooler) and will be responsible for shipping samples from the field to the laboratory. The sampler will provide the project number, the CLP case or SAS number, and the sampler's signature as header information on the chain-of-custody record. The common name of the site will not be included in this form or other sample documentation because CLP laboratories may perform analyses for responsible parties associated with the site. For each station number, the sampler will indicate the date, time, sample status (composite or grab sample), station location, number of containers, analytical parameters, EPA sample numbers, and CLP sample numbers. When shipping the samples, the sampler will sign the bottom of the form and enter the date and time (military) that the samples were relinquished. The sampler will enter the carrier name and air bill number under the "Remarks" section on the bottom right of the form. The original signature copy of the chain-of-custody record will be enclosed in a plastic bag (along with any other necessary CLP or EPA sample documentation) and secured to the inside of the cooler lid. A copy of the custody record will be retained for PRC files.

Shipping coolers will be secured for shipment by placing custody seals across all four sides of the cooler lid. Commercial carriers will not be required to sign off on the chain-of-custody forms provided that the forms are sealed inside the shipping cooler and the custody seals remain intact.

5.2 LABORATORY CUSTODY PROCEDURES

The EPA Region 10 Manchester Laboratory or CLP laboratories performing the chemical analyses will be responsible for following all CLP-required chain-of-custody procedures presented in the CLP SOWs (EPA, 1990a and 1990b).

FIGURE 5-1

EXAMPLE OF A TYPICAL SAMPLE TAG AND CUSTODY SEAL

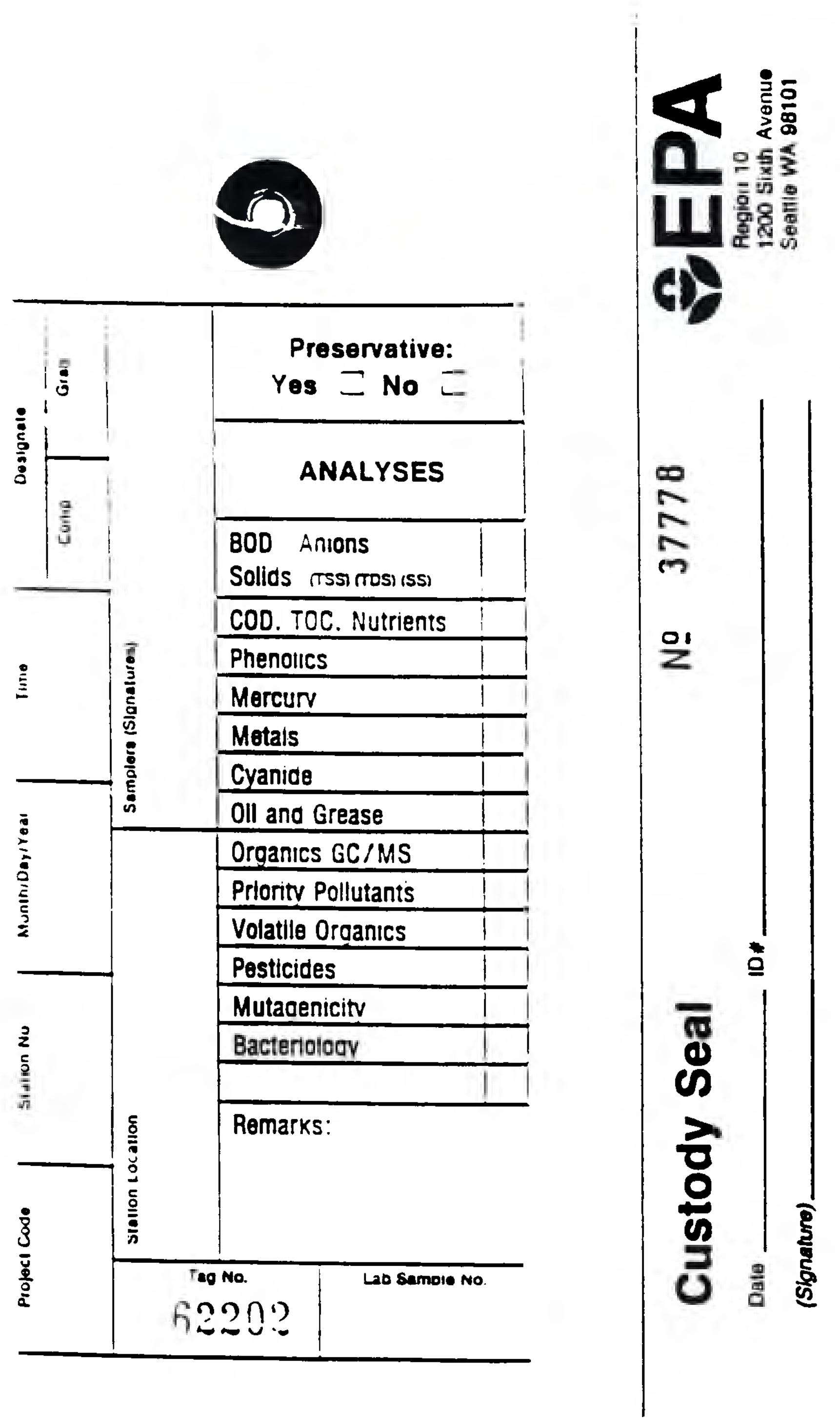


FIGURE 5-2

EXAMPLE OF AN ORGANIC SAMPLE ANALYSIS REQUEST FORM

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Organic Traffic Report
(For CLP Use Only)

Case Number | SAS No. (if applicable)

1. Type of Activity (Check one)
   - ENF
   - ER
   - ESI
   - NPLD
   - O&M
   - PA
   - RA
   - RD
   - RIFS
   - SI
   - ST
   - STPA
   - STSI
   - Other *(Specify)*

Non-Superfund Program

Site Name

City, State

Site Spill ID

2. Region Number | Sampling Co.

Sampler (Name)

3. Ship To:

4. Date Shipped | Airbill Number

Carrier

Triple volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

5. Sample Description *(Enter in Column A)*
   1. Surface Water
   2. Ground Water
   3. Leachate
   4. Rinsate
   5. Soil/Sediment
   6. Oil (SAS)
   7. Waste (SAS)
   8. Other (SAS) *(Specify)*

CLP Sample Number *(From labels)*	(A) Sample Description *(From box 5)*	(B) Concentration L=low M=med H=high	(C) RAS Analysis VOA	BNA	Pest/PCB	(D) Special Handling	(E) Station Location	(F) Date/Time of Sample Collection	(G) Corresponding CLP Inorganic Sample Number

EPA Form 9110-2 (8-88) Replaces EPA Form 2075-7, which may be used. Blue - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

FIGURE 5-3
EXAMPLE OF AN INORGANIC SAMPLE ANALYSIS REQUEST FORM

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Inorganic Traffic Report
(For CLP Use Only)

Case Number | SAS No. (if applicable)

1. Type of Activity (Check one)
☐ ENF ☐ NPLD ☐ RA ☐ SI ☐ STSI
☐ ER ☐ O&M ☐ RD ☐ ST ☐ Other *(Specify)*
☐ ESI ☐ PA ☐ RIFS ☐ STPA

Non-Superfund Program

Site Name

City, State | Site Spill ID

2. Region Number | Sampling Co.

Sampler (Name)

3. Ship To:

4. Date Shipped | Airbill Number

Carrier

Double volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

5. Sample Description *(Enter in Column A)*

1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) *(Specify)*

CLP Sample Number *(From labels)*	(A) Sample Description *(From box 1)*	(B) Concentration L=low M=med H=high	(C) RAS Analysis		(D) Special Handling	(E) Station Location	(F) Date/Time of Sample Collection	(G) Corresponding Organic Sample Number
			Total Metals	Cyanide				

EPA Form 9110-1 (8-88) Replaces EPA Form 2075-6, which may be used. **Green - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy**

FIGURE 5-4
EXAMPLE OF A SPECIAL ANALYTICAL SERVICES REQUEST FORM

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office:	Sampling Date(s):	Ship To:	For Lab Use Only
Sampling Contact:	Date Shipped:		Date Samples Rec'd:
(name)	Site Name/Code:		Received By:
(phone)		Attn:	

	Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			
			For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

FIGURE 5-5

EXAMPLE OF AN EPA REGION 10 LABORATORY ANALYSIS REQUEST FOR ORGANICS

EPA Region 10 Laboratory

Analyses Required

PRIORITY POLLUTANTS - ORGANICS

Project Name: ____________ Project Code: ____________ Account Code: ____________

Matrix Codes *(circle one only)*

10 Water-Total
11 Water-Dissolved
40 Sediment/Soil
45 Semi-Solid/Sludge
46 Sediment for EP Toxicity
70 Tissue
80 Oil/Solvent
00 Other

Sample Numbers

Analy/Comp Init/Date

GC/MS Organics

51	Volatile Organics	VOA
62	Base/Neutrals Only	B/N
65	Acids Only	Acid
68	Base/Neutrals/Acids	B/N/A
60	Specific (GC/MS) Organics	list below

(additional space to list is provided below)

GC Organics

42	EDB	EDB
[illegible]	Trihalomethanes	Trihal
54	Purgeable halocarbons	Purg
71	Pesticide/PCB's	Pest/PCB
72	Pesticides Only	Pest
73	Herbicides *(List each/circle on reverse)*	
74	PCB's Only	PCB
77	Organophosphate Pesticides	*(List each)*
70	Specific Pesticides	*(List each)*
80	Specific (GC/MS) Organics	*(List each)*

(additional space to list is provided below)

Specific Organics/Other Miscellaneous

40	PolyAromHydro (HPLC)	PAH
67	Oil Identification	Oil-Id

(additional space to list is provided below)

Save samples after analysis? NONE †SOME †ALL *(if SOME, circle sample numbers)*

List any additional specific organics:

Special detection Limits and comments:

Requester's Signature

†If samples should be saved, why?

EPA X-81 Lab Copy

FIGURE 5-6
EXAMPLE OF AN EPA REGION 10 LABORATORY ANALYSIS REQUEST FOR METALS

EPA Region 10 Laboratory **Analyses Required**

METALS

Project Name: ____________ Project Code: ____________ Account Code: ____________

Matrix Codes *(circle one only)*
10 Water-Total
11 Water-Dissolved
40 Sediment/Soil
45 Semi-Solid/Sludge
46 Sediment for EP Toxicity
70 Tissue
80 Oil/Solvent
00 Other

Metals
(circle WG# and elements requiring analysis below)
Workgroup 30 - Standard Method
Workgroup 34 - EP Toxicity Method
Workgroup 47 - TCLP Method

Element	Symbol	Sample Numbers	Analy/Comp Init/Date
Aluminum	Al		
Antimony	Sb		
Arsenic	As		
Barium	Ba		
Beryllium	Be		
Cadmium	Cd		
Calcium	Ca		
Chromium	Cr		
*Chromium Hexavalent	Cr+6		
Cobalt	Co		
Copper	Cu		
Iron	Fe		
Lead	Pb		
Magnesium	Mg		
Manganese	Mn		
Mercury	Hg		
Molybdenum	Mo		
Nickel	Ni		
Potassium	K		
Selenium	Se		
Silver	Ag		
Sodium	Na		
Thallium	Tl		
Vanadium	V		
Zinc	Zn		

Save samples after analysis? NONE †SOME †ALL *(if SOME, circle sample numbers)*

* The Laboratory requires advance notice prior to receiving samples for Cr+6.

Special Limits, methods and comments: ____________

Requester's Signature ____________ *Date* ____________

†If samples should be saved, why? ____________

Note: Any Element may be determined by AA or ICP

EPA X-92 Lab Copy

FIGURE 5-7
EXAMPLE OF A CHAIN-OF CUSTODY RECORDING FORM

FIELD SAMPLE DATA AND CHAIN OF CUSTODY SHEET

EPA Region 10
1200 Sixth Avenue
Seattle WA 98101

Case No.: ____________ ☐ Enforcement/Custody Miscellaneous: ____________ Sampling Crew: ____________

Project Code: ________ Account: ________ ☐ Data Confidential

Name/Location ____________ (EPA Lab Only. Leave Blank for Contract Lab) ☐ Possible Toxic/Hazardous

Proj. Off.: ____________ Tel.# ________ ☐ Data for Storet

Recorder: ____________ (Signature Required)

SOURCE CODE	MATRIX					# CONTAINERS							LAB NUMBER			STORET STATION NUMBER	SAMPLING DATE & TIME				TRAFFIC REPORT NUMBERS		SAMPLER'S INITIALS	STATION DESCRIPTION
	Oil	Water	Sediment	Tissue	[illegible]	Qt. Cubit	Gal Cubit	16 oz	8 oz	120 ml	40 ml	Other	Yr	Wk	Seq		Yr	Mo	Dy	Time	Org	Inorg		

LAB NUMBER			DEPTH	Units	Type	COL MTO CD	QA CODE	TEMP DEG C	pH	CNDCTVTY umho/cm	COMPOSITE ONLY				
											ENDING DATE				
Yr	Wk	Seq									Mo	Dy	Time	Type	Freq

CONDITION OF SAMPLES UPON RECEIPT AT LAB

CUSTODY SEALS INTACT yes ☐ no ☐ none

CHAIN OF CUSTODY RECORD

RELINQUISHED BY (Signature)	RECEIVED BY (Signature)	DATE/TIME
RELINQUISHED BY (Signature)	RECEIVED BY (Signature)	DATE/TIME
RELINQUISHED BY (Signature)	RECEIVED BY (Signature)	DATE/TIME
RELINQUISHED BY (Signature)	RECEIVED BY MOBILE LAB FOR FIELD ANALYSIS (Signature)	DATE/TIME
DISPATCHED BY (Signature) DATE/TIME	RECEIVED FOR LAB BY (Signature)	DATE/TIME
METHOD OF SHIPMENT		

Field or Office Copy GPO 1988-691-384/1003

6.0 CALIBRATION PROCEDURES AND FREQUENCY

Both laboratory and field equipment must be calibrated on a regular basis to assure the accuracy of analyses. This section describes the calibration procedures and frequency for measuring and testing equipment.

6.1 FIELD EQUIPMENT

PRC personnel will use an HNu model P-101 photoionization detector (PID) to monitor ambient air conditions for health and safety precautions at the RBT site. The operator's manual for the unit is included as Appendix B. Before transport to the field, the battery and fan of the HNu will be checked to assure that they are operational, and the unit will be calibrated. The calibration will be performed with isobutylene gas supplied by the manufacturer. The unit will be calibrated in the field before use to assure that damage has not occurred during transportation. All calibration information, including date, time, pressure of calibration gas, span setting of the HNu, and name of the equipment operator, will be recorded in the project logbook.

6.2 LABORATORY EQUIPMENT

Laboratory calibration requirements for CLP RAS procedures can be found in the EPA CLP SOWs for organic and inorganic analysis (EPA, 1990a and 1990b). Laboratory calibration procedures for CLP SAS procedures for PAH, chlorophenols, arsenic, and chromium are specified in the SAS request forms included in Appendix A.

7.0 ANALYTICAL PROCEDURES

This section presents a discussion of the field and laboratory analytical procedures that will be used during the O&M.

7.1 FIELD ANALYTICAL PROCEDURES

PRC will perform ambient air monitoring with an HNu PID. The HNu can detect a variety of VOCs but cannot identify discrete compounds unless directly calibrated. The HNu does, however, measure individual VOCs relative to isobutylene (refer to the HNu owner's manual in Appendix B). The HNu provides an approximate measurement of total VOCs. PRC will use the HNu to screen for the presence of total VOCs that may produce a health and safety hazard.

7.2 LABORATORY ANALYTICAL PROCEDURES

The EPA Region 10 Manchester Laboratory or a CLP laboratory, depending on availability, will be used for analytical support for the O&M. The levels of precision and accuracy specified in the most recent CLP SOWs for organic and inorganic analysis (EPA, 1990a and 1990b) will serve as standard DQOs for the CLP RAS procedures. CLP SAS will be requested for samples analyzed for PAH, chlorophenols, arsenic, and chromium. The procedures are specified in the SAS request forms included in Appendix A.

8.0 INTERNAL QUALITY CONTROL CHECKS

An internal QC system is a set of routine internal procedures to promote data output of a measurement system that meets the objectives prescribed in the data QA/QC program. Inherent and implied in this control function is a parallel function of measuring and defining the quality of the data output. A well designed internal QC program must be capable of controlling and measuring the quality of the data in terms of precision and bias. Precision reflects the influence of the inherent variability in any measurement system. Bias represents a consistent error in the measurement system.

For samples received at the RBT site, PRC will use the internal QC measures described in the following sections to assure a high degree of precision and accuracy.

8.1 FIELD QUALITY CONTROL CHECKS

As a QC check on field sampling, PRC will receive field duplicate samples, equipment rinsate field blanks, trip blanks and field (transfer) blanks to be sent to the laboratory at specified frequencies discussed in Section 3.4.

Field QC checks also include regular and continuing calibration of all field measuring equipment. This field equipment will include an HNu model P101 PID used to monitor for volatile organic vapors. Calibration procedures for the HNu are discussed in Appendix B.

8.1.1 Field Duplicates Samples

A field duplicate is defined as two or more samples collected (received) independently at a sampling location during a single act of sampling. The total number of field duplicates for each analysis is presented in Section 4.0. Duplicates will be received at a minimum frequency of one per system (monitoring well and leachate collection). Duplicate sample containers will be received alternately between the environmental sample and duplicate sample.

Field duplicates will be identified so that the laboratory cannot distinguish them from other samples. Therefore, one complete sample set will be identified with a "coded" or false identifier in the same format as other identifiers used for this sample matrix. Both the coded and the true identifiers will be recorded in the field notebook. On chain-of-custody forms, the coded identifier will be used. These coded field duplicates will be used to assess the representativeness of the sampling procedure as well as laboratory analytical precision.

8.1.2 Equipment Rinsate Field Blanks

An equipment rinsate field blank is a sample received in the field by pouring carbon-free water over the decontaminated ground-water sampling equipment and into the appropriate containers. The equipment rinsate field blank will be shipped to the laboratory for analysis along with the other environmental samples. The equipment rinsate field blanks will determine whether sampling equipment was sufficiently decontaminated to prevent cross-contamination between samples. Equipment rinsate field blanks will be received at a minimum frequency of one per system (monitoring well and leachate collection) as indicated in Section 4.0. The equipment rinsate field blanks will be analyzed for all laboratory-measured parameters.

8.1.3 Trip Blanks

A trip blank consists of sample containers (two 40-ml VOA vials) filled with carbon-free water by the EPA Region 10 Manchester laboratory. The trip blank will be carried into the field and handled like a sample but not opened. It will be returned to the laboratory for analysis along with the other environmental samples. The trip blanks will be analyzed only for VOCs and will be used to determine if contaminants have been introduced during sample handling and shipment. One trip blank will be included with each shipment of VOC samples sent to the laboratory.

8.1.4 Field (Transfer) Blanks

A field (transfer) blank consists of sample containers (two 40-ml VOA vials) filled with carbon-free water in the field by PRC personnel. The field (transfer) blanks will be returned to the laboratory for analysis along with the other environmental samples. The field (transfer) blanks will be analyzed only for VOCs and will be used to determine if contaminants have been introduced from ambient conditions during sample collection (receiving). One field (transfer) blank will be prepared per day of sampling.

8.2 LABORATORY QUALITY CONTROL CHECKS

QC data are necessary to determine precision and accuracy of analyses and to demonstrate the absence of interferences and contamination of glassware and reagents. The CLP RAS methods include the use of laboratory blanks, MS/MSD samples, initial and continuing calibrations, and other similar measures as specified in the CLP SOW for organic analysis (EPA, 1990a). Laboratory quality control checks for SAS analyses are specified in the SAS request forms included in Appendix A.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

The data reduction, validation, and reporting process includes all steps between the original instrument or visual reading and the final complete report. Data reduction includes laboratory calculations for unit conversions, dilutions, and similar factors and preparation of the final report. To validate the data, someone other than the laboratory analyst reviews the data reduction procedures to determine the acceptability of the data and any necessary qualifiers. Reporting includes transcribing these validated data into a final report and interpreting them. Data reduction and data validation differ among analytical methods, but the reporting process is common to all data.

9.1 DATA REDUCTION

The EPA Region 10 Manchester Laboratory and CLP laboratories performing RAS analyses will be required to follow data reduction procedures according to EPA Laboratory Data Functional Guidelines for Evaluation of Organics (EPA, 1988b) and Inorganics (EPA, 1988c) Analyses. Data reduction for the SAS analyses is specified in the SAS request forms included in Appendix A.

Field parameters to be measured during the O&M split sampling will include volatile organic vapors in the air. All field parameters will be measured by direct reading of instruments. Results will be recorded directly into filed notebooks, thus no data reduction is required.

9.2 DATA VALIDATION

This section outlines data validation procedures for both field and laboratory measurements.

9.2.1 Field Measurements

All field data will be generated by qualified field personnel and immediately entered in a field logbook. These data will be reviewed daily for completeness, consistency, and proper procedures (such as calibration) by the field team leader.

9.2.2 Laboratory Measurements

An independent data validation of 100 percent of CLP RAS and SAS raw data will be performed by PRC personnel not currently involved in this project. Validation of the CLP RAS data will be carried out according to EPA Laboratory Data Functional Guidelines for Evaluation of Organics (EPA, 1988b) and Inorganics Analyses (EPA, 1988c). SAS data will be validated based upon requirements outlined in the SAS request forms included in Appendix A.

For samples analyzed by the EPA Region 10 Manchester Laboratory, EPA will perform the data validation of 100 percent of all raw data. The data will be screened by EPA for precision and accuracy according to the same organic and inorganic functional guidelines cited above. SAS data will be validated based upon requirements outlined in the SAS Request Form in Appendix A.

9.3 REPORTING

All data from EPA Region 10 Manchester Laboratory and CLP RAS laboratories will be reported in a standard CLP RAS data deliverable format. Reporting requirements for the CLP SAS analysis are specified in the SAS Request Form in Appendix A.

All data generated in the field will be collected in a project file at the PRC Seattle office. All laboratory reports and other data will also be placed in this file. This file will be organized to allow ready identification and retrieval of any desired information.

Quantitative information will be entered into databases that will be printed out, checked against the original data sheets, and corrected before use. The resulting databases will be supplemented by the text of the O&M report, including data interpretations. All PRC deliverables are reviewed by a technical editor, technical reviewer, and QC coordinator before release.

10.0 PERFORMANCE AND SYSTEMS AUDITS

All laboratory and field work conducted for the RBT O&M may be subject to performance and systems audits. Performance audits check the operation of a specific study component, such as a sampling method or an analytical procedure. Systems audits are broader and include a thorough evaluation of both laboratory and field QA/QC methods, such as data validation procedures, corrective action procedures, or sample custody procedures. Audits may be internal (conducted by PRC QA/QC personnel within the organization being audited) or external (conducted by EPA or another outside agency).

Audits are randomly scheduled by QA/QC personnel and are generally not announced beforehand. If QA/QC personnel find what seems to be a systematic problem with a particular component of the sampling and analysis program, they will normally perform a series of audits on related activities to identify and correct the problem. Audit results are incorporated into the project reporting system, normally in the monthly report.

10.1 LABORATORY AUDITS

Performance and systems audits of CLP RAS and SAS laboratories are the responsibility of the EPA. Specific details are included within CLP documents (such as the CLP SOWs) and in each laboratory's standard operating procedures (SOPs). If required, internal audits will be conducted by personnel from CLP laboratories performing the analyses. External audits, if required, are usually performed by the EPA Environmental Monitoring Systems Laboratory (EMSL) in Las Vegas. However, evidence audits of CLP laboratories may also be conducted by the National Enforcement Investigations Center (NEIC) in Denver.

10.2 FIELD AUDITS

Internal performance and systems audits of all PRC field activities at RBT will be coordinated by the PRC TES 12 QA manager, Dave Liu, in accordance with TES 12 QA Program Plan (PRC, 1988). If a field audit is scheduled to be conducted for the RBT O&M, a site-specific audit checklist will be prepared (Figure 10-1). This checklist will be based on information contained in the QAPjP and health and safety plan. Using the checklist, auditors will evaluate whether field personnel are operating in compliance with procedures specified in these plans, including the following:

- Initial and continuing equipment calibration
- Field measurements
- Sample collection (receiving)
- Sample labeling, handling, and custody
- Data collection and record keeping
- Health and safety monitoring
- Logbook completeness
- Photographic documentation
- Availability of documents used to evaluate RBT's compliance

External field audits for this project are the co-responsibility of the EPA Region 10.

FIGURE 10-1
AUDIT REPORT FORM

PRC Environmental Management, Inc.

Audit Report

QA/QC Level _____

Project/Contract No.: _______________

Work Assignment No: _______________

Work Assignment Manager: _______________

Region: _______________

Firm: _______________

Date of Audit: _______________

Auditor: _______________

Brief Description of Work Assignment:

____

____

____

Audit Summary:

____

____

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Corrective Action Required:

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Remarks:

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Auditor Signature: _______________________

Date: _______________

Distribution: 1) Original to project file 2) Copy to QA/QC file 3) Copy to auditor

11.0 PREVENTATIVE MAINTENANCE

Preventative maintenance (PM) includes inspecting, repairing, and adjusting equipment and instruments before any deficiencies have a significant effect on performance. These techniques are a necessary part of the procedures for carrying out a particular operation with a particular type of equipment.

11.1 LABORATORY EQUIPMENT

The EPA Region 10 Manchester Laboratory or the CLP RAS laboratory that analyzes the ground-water and leachate samples will follow necessary PM actions detailed in its internal SOPs as well as PM required by the CLP SOWs. These include (1) tuning and calibration (both initial and continuing) of machines, (2) use of internal standards, and (3) related activities such as corrective action. Details of these requirements are included in the CLP SOW for organic analyses (EPA, 1990a). PM requirements for SAS analyses are specified in the SAS request forms included in Appendix A.

11.2 FIELD EQUIPMENT

PRC will perform regular preventative maintenance of all field equipment. All field monitoring and analytical equipment will be maintained in accordance with the manufacturers' recommended schedules and procedures. Field personnel will maintain records of service, calibration, and use. Instrument problems encountered in the field will be detailed in the field logbook and dealt with on-site, if possible.

The primary PM technique for field analyses is the preliminary calibration of equipment. As detailed in the HNu operator's manual (Appendix B), this typically includes a battery check, zero adjustment, and linearity (or high end) adjustment. If the instrument cannot be correctly calibrated, it will be disassembled, cleaned, reassembled, and recalibrated.

12.0 PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The QA/QC objectives described in Section 3.0 are goals necessary to satisfactorily complete the RBT O&M. This section discusses the means for assessing whether those objectives have been met. The assessment is part of the data reduction and validation process discussed in Section 9.0.

12.1 LABORATORY RESULTS

The precision of CLP RAS and SAS laboratory results will be determined primarily by calculating the relative percent difference (RPD) for duplicate samples. These will include field duplicates, laboratory duplicates, and MS/MSD samples. The laboratory will determine the accuracy of results by calculating percent recovery values for MS/MSD samples. In addition, the laboratory will use laboratory blanks, calibration standards, and internal standards to establish analytical accuracy, as detailed in the CLP SOW. Completeness of all laboratory results will be determined by comparing the number of validated, usable results to the number of samples planned.

12.2 CALCULATIONS

The primary statistic used for estimating precision is RPD for duplicate measurements. RPD is calculated as follows:

$$RPD = \frac{|X_1 - X_2|}{(X_1 + X_2)/2} \times 100 \qquad (12\text{-}1)$$

where X_1 and X_2 are the results of duplicate measurements and $|X_1 - X_2|$ is the absolute value of the difference in the two measurements.

If there are three or more replicates, the relative standard deviation (%RSD) will be calculated as a measure of precision:

$$\%RSD = (SD/\bar{X}) \times 100 \qquad (12\text{-}2)$$

where $\bar{X}$ is the average of the data points (X_1, X_2, . . . X_n) and SD is the standard deviation of the individual measurements.

Accuracy can be estimated by calculating the percent difference (%D) between an instrument response and a known standard:

$$\%D = (S-X)/S \times 100 \qquad (12\text{-}3)$$

where S is the concentration of a known standard and X is the measured instrument response. This determination of accuracy can be used for both laboratory and field measurements.

Alternatively, accuracy can be measured as the percent recovery (%R) from the analytical results of surrogate or analyte compounds spiked into a sample:

$$\%R = (M-N)/S \times 100 \qquad (12\text{-}4)$$

where M is the measured analyte concentration in the spiked sample, N is the concentration of the analyte in the original sample, and S is analyte concentration spiked into the original sample. This measurement of accuracy is most appropriate for laboratory results.

Percent completeness (%C) is a measure of (1) the number of samples actually received compared to the number of samples required for characterization or (2) the amount of valid data obtained compared to the amount of data expected under normal conditions. In most cases, the "number of samples required for characterization" and the "amount of data expected under normal conditions" are the same as the number of samples planned, N. Thus, percent completeness can be defined as follows:

$$\%C = V/N \times 100 \qquad (12\text{-}5)$$

where V is the number of valid results and N is the total number of samples planned.

Percent completeness can also be measured as the percent of samples planned that were actually received:

$$\%C = C/N \times 100 \qquad (12\text{-}6)$$

where C is the number of samples received and N is the total number of samples planned.

13.0 CORRECTIVE ACTION

Corrective action must be initiated whenever a system is not functioning properly. These situations may be identified during performance or system audits or by the analysts themselves. Corrective action may take place in the laboratory or in the field.

13.1 LABORATORY CORRECTIVE ACTION

EPA Region 10 and or CLP laboratory analyses will be conducted for ground-water and leachate samples. If QC audits conducted by EPA identify a noncompliance situation, the problem will be reported to the WAM. Major noncompliance situations within the CLP laboratory are usually handled between the laboratory, the CLP Sample Management Office, and EPA Region 10. Detailed procedures for corrective action during RAS sample analyses are provided in the CLP SOWs. For SAS analyses, the CLP laboratory will be required to follow its own internal corrective action procedures. However, if corrective action beyond the scope of the SAS request is required, the laboratory will advise the CLP Sample Management Office (SMO), and the SMO will advise the EPA Region 10 RSCC. The RSCC, SMO, and laboratory will then determine the appropriate corrective action.

Frequently, problems with EPA Region 10 and CLP analyses result from matrix effects, that make results questionable (estimates, qualified as "J") or unusable (rejected, qualified as "R"). The Region 10 CRL, WAM, PRC project manager, and PRC TES 12 QA manager will jointly determine the acceptability of data and determine appropriate corrective action. Corrective actions may include the following:

- Reanalyzing samples if holding time criteria permit
- Resampling and analyzing the samples
- Evaluating and amending sampling and analytical procedures
- Accepting data and acknowledging a level of uncertainty

13.2 FIELD CORRECTIVE ACTION

During field investigations, any problems that affect receiving samples and monitoring data will be documented and recorded in a field logbook by the person who identified the problem. Serious problems that affect overall project objectives will be brought to the attention of the PRC site manager. The project manager will complete a Corrective Action Request Form (Figure 13-1) and immediately notify the PRC TES 12 QA manager. The PRC TES 12 QA manager, project manager, or their designees are responsible for identifying the causes of the problems and developing solutions.

FIGURE 13-1
CORRECTIVE ACTION REQUEST FORM

PRC Environmental Management, Inc.

Corrective Action Request Form QA/QC Level _____

Project/Contract No.: ______________________

Work Assignment Number: ______________________

Site Location: ______________________

Firm: ______________________

To (Work Assignment Manager): ______________________

From (Reviewer): ______________________
Signature

Date: ______________________

Description of Problem: ______________________

____

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____

Corrective Action Requested: ______________________

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The above corrective action must be completed by: ______________________
(Date)

Corrective Action Taken: ______________________

____

____

____

FIGURE 13-1 (continued)
CORRECTIVE ACTION REQUEST FORM

QA/QC Level ____

Work Assignment Manager:

(Subcontractor QA Manager)

Acknowledgement of Receipt

____
(Initial/Date)

Correction Action Completed

____
(Initial/Date)

Reviewer

Corrective Action is/is not satisfactory

____
(Initial/Date)

Remarks

____
____
____
____

QA/QC Coordinators:

Corrective Action is/is not satisfactory

____
(Initial/Date)

Remarks

____
____
____
____

Distribution: 1) Original to project file 2) Copy to QA/QC file 3) Copy to reviewer

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Effective management of environmental measurements requires timely assessment and review of activities. This requires interaction among PRC personnel collecting the data, the PRC project manager, the PRC Regional Manager, the PRC TES 12 program manager, and EPA personnel. Written reports of field activities may be necessary to provide an on-going evaluation of measurement data quality. These reports are produced on an as-required basis and may include the following:

- QA/QC audit results and other inspection reports
- Summary of corrective action activities, including any unresolved problems or past-due corrective actions
- Summary of unscheduled equipment maintenance activities
- Summary of any QAPjP changes
- Summary of project QA/QC activities and status

Reports of this type will be distributed to the PRC project manager, Regional Manager, TES 12 QA manager, and EPA WAM.

Routine QA/QC reports for TES 12 (contract number 68-W9-0009) are prepared by the PRC Regional Manager and submitted to the PRC TES 12 QA manager (PRC, 1988). If significant QA/QC activities concerning the RBT O&M appear in this program QA/QC report, these QA/QC activities will also be described in the monthly progress reports for the RBT O&M project.

PRC will submit a report at the completion of the field investigation planned for the RBT O&M. The report will contain a separate QA/QC section summarizing data quality information and identifying any significant QA/QC activities that occurred during the investigation.

15.0 REFERENCES

EPA, 1983. Methods for Chemical Analysis of Water and Wastes, Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency. EPA-600/4-79-020. March 1983.

EPA, 1986a. Test Methods for Evaluating Solid Waste, SW-846. Third Edition, November 1986.

EPA, 1986b. National Enforcement Investigations Center Policies and Procedures. EPA-300/9-78-001-R.

EPA, 1986c. Consent Agreement and Final Order. RCRA Docket No. 1085-09-26-3008P. November 21, 1986.

EPA, 1986d. RCRA Ground-Water Monitoring Technical Enforcement Guidance Document. Office of Solid Waste and Emergency Response (OSWER) 9950.1. September 1986.

EPA, 1988a. User's Guide to the Contract Laboratory Program.

EPA, 1988b. Laboratory Data Validation Functional Guidelines for Evaluation of Organic Analyses. Hazardous Site Evaluation Division, February.

EPA, 1988c. Laboratory Data Validation Functional Guidelines for Evaluation of Inorganic Analyses. Hazardous Site Evaluation Division, July.

EPA, 1988d. Final Operation and Maintenance Inspection Guide (RCRA Ground-Water Monitoring Systems), Office of Waste Programs Enforcement, U.S. Environmental Protection Agency, OSWER 9950.3. March 1988.

EPA, 1990a. Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, Document Number OLM01.0.

EPA, 1990b. Contract Laboratory Program Statement of Work for Inorganics Analysis, Multi-Media; Multi-Concentration, Document Number ILM01.0.

PRC, 1988. Quality Assurance Program Plan, TES 12 contract, March 1988.

PWT, 1988. Ground-Water Monitoring Report for the RBT Landfill Site, Ridgefield, Washington, prepared for Pacific Wood Treating Corporation by David J. Newton Associates, Inc., Portland, Oregon. December 28, 1988.

Sax, N.I. and R.J. Lewis, 1987. Hawley's Condensed Chemical Dictionary, Eleventh Edition. Van Nostrand Reinhold, New York, NY.

Tetra Tech, 1989. RCRA Comprehensive Ground-Water Monitoring Evaluation, Ridgefield Brick and Tile/Pacific Wood Treating, Ridgefield, Washington, September 1989.

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request

[X] Regional Transmittal   [ ] Telephone Request

A. EPA Region/Client: Region 10

B. Authorized By: Bruce Woods (206) 553-1193

C. Prepared By: Laura CastrillI (206) 553-4323

D. Date of Request: March 8, 1991

E. Site Name: Ridgefield Brick and Tile (RBT)
Ridgefield, Washington

F. 2 digit Superfund site identifier: N/A

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   The requested analytical service will be used to determine the concentration of polynuclear aromatic hydrocarbons (PAH) in ground water (System No. 1) and leachate (System No. 2) at RBT.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):

   This will be a split sampling event.

   Six aqueous environmental samples, two aqueous duplicate samples, and two aqueous equipment rinsate blanks (**10 samples/blanks total**) will be received to analyze for PAH. From previous sampling data, these samples and blanks are expected to contain low concentrations of PAH.

3. **Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):**

   The purpose of this analysis will be to supply supporting analytical data to EPA to aid in characterizing the RBT site. EPA is considering a clean closure option for the RBT landfill.

4. **Estimated date(s) of collection:** March 27 and 28, 1991

5. **Estimated date(s) and method of shipment:**

   Samples will be shipped by Federal Express on March 27 and 28, 1991 following each day's sampling activities.

6. **Number of days analysis and data required after laboratory receipt of samples:**

   Samples must be preserved with 1.5 mL of 10% $Na_2S_2O_3$ per liter and stored in the dark at 4°C. The laboratory must extract these samples within seven days of collection. Analysis must follow within 40 days of extraction.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

   The protocols to be used for PAH extraction are taken from Test Methods for Evaluating Solid Waste (SW-846). Samples must be extracted by Method 3520 and analysis performed using Method 8310. If interferences are present after initial analyses, clean up will be required using Method 3620. Copies of these methods from SW-846 are attached to this SAS request.

8. **Special technical instructions (if outside protocol requirements, specify compound names, SAS numbers, detection limits, etc.):**

   No special techniques will be required other than specified by SW-846 Methods 3520/3620/8310.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.**

   Data will be reported in the standard Contract Laboratory Program Routine Analytical Service format.

11. **Name of sampling/shipping contact:**

   Gary Bruno
   PRC Environmental Management, Inc.

   Phone: 206/624-2692

12. **Data Requirements**

Parameter	Quantitation Limit	Precision (percent or Concentration)
PAH	0.013 - 2.3μg/L*	± 20

* See Method 8310 for each compound and its detection limit.

13. **QC Requirements**

Audits required	Frequency of Audits	Limits (percent or Concentration)
Laboratory Control Samples	Once per 20 Samples or once per 12 hours of continuous assay.	± 10 %
Analytical System Preparation	See Method 8310, Sections 8.0 and Method 8000, Section 8.6. (Copies are attached to this SAS request).	See Method 8310 Table 3 and Method 8000 Section 8.6.
MS/MSD	Once per 20 samples	± 30 %
Laboratory Duplicates	Once per 20 samples	± 20 %

If interferences are found in samples and Method 3620 must be applied, then all associated quality control samples must also be processed through this clean up method. Additionally, before Method 3620 may be applied, the analyst should show that the compounds of interest are being quantitatively recovered before applying this method to actual samples (see 3620, Section 8.0).

14. **Action Required if Limits are Exceeded**

Recalibrate analysis instrument as indicated and reassay or call Bruce Woods, QA chemist, at 206-553-1193 (FTS 399-1193) or Gerald Muth, CLP DPO, at 206-871-0748 (FTS 390-1282), immediately, for problem resolution.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

METHOD 8310

POLYNUCLEAR AROMATIC HYDROCARBONS

1.0 SCOPE AND APPLICATION

1.1 Method 8310 is used to determine the concentration of certain polynuclear aromatic hydrocarbons (PAH) in ground water and wastes. Specifically, Method 8310 is used to detect the following substances:

Acenaphthene	Chrysene
Acenaphthylene	Dibenzo(a,h)anthracene
Anthracene	Fluoranthene
Benzo(a)anthracene	Fluorene
Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
Benzo(b)fluoranthene	Naphthalene
Benzo(ghi)perylene	Phenanthrene
Benzo(k)fluoranthene	Pyrene

1.2 Use of Method 8310 presupposes a high expectation of finding the specific compounds of interest. If the user is attempting to screen samples for any or all of the compounds listed above, he must develop independent protocols for the verification of identity.

1.3 The method detection limits for each compound in reagent water are listed in Table 1. Table 2 lists the practical quantitation limit (PQL) for other matrices. The sensitivity of this method usually depends on the level of interferences rather than instrumental limitations. The limits of detection listed in Table 1 for the liquid chromatographic approach represent sensitivities that can be achieved in the absence of interferences. When interferences are present, the level of sensitivity will be lower.

1.4 This method is recommended for use only by experienced residue analysts or under the close supervision of such qualified persons.

2.0 SUMMARY OF METHOD

2.1 Method 8310 provides high performance liquid chromatographic (HPLC) conditions for the detection of ppb levels of certain polynuclear aromatic hydrocarbons. Prior to use of this method, appropriate sample extraction techniques must be used. A 5- to 25-uL aliquot of the extract is injected into an HPLC, and compounds in the effluent are detected by ultraviolet (UV) and fluorescence detectors.

2.2 If interferences prevent proper detection of the analytes of interest, the method may also be performed on extracts that have undergone cleanup using silica gel column cleanup (Method 3630).

3.0 INTERFERENCES

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.2 Interferences coextracted from the samples will vary considerably from source to source. Although a general cleanup technique is provided as part of this method, individual samples may require additional cleanup approaches to achieve the sensitivities stated in Table 1.

3.3 The chromatographic conditions described allow for a unique resolution of the specific PAH compounds covered by this method. Other PAH compounds, in addition to matrix artifacts, may interfere.

4.0 APPARATUS AND MATERIALS

4.1 Kuderna-Danish (K-D) apparatus:

4.1.1 **Concentrator tube:** 10-mL, graduated (Kontes K-570050-1025 or equivalent). Ground-glass stopper is used to prevent evaporation of extracts.

4.1.2 **Evaporation flask:** 500-mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs.

4.1.3 **Snyder column:** Three-ball macro (Kontes K-503000-0121 or equivalent).

4.1.4 **Snyder column:** Two-ball micro (Kontes K-569001-0219 or equivalent).

4.2 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.3 Water bath: Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

4.4 Syringe: 5-mL.

4.5 High pressure syringes.

4.6 HPLC apparatus:

4.6.1 **Gradient pumping system:** Constant flow.

4.6.2 **Reverse phase column:** HC-ODS Sil-X, 5-micron particle size diameter, in a 250-mm x 2.6-mm I.D. stainless steel column (Perkin Elmer No. 089-0716 or equivalent).

5.5 Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

5.5.1 Prepare calibration standards at a minimum of five concentration levels for each analyte as described in Paragraph 5.4.

5.5.2 To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with acetonitrile.

5.5.3 Analyze each calibration standard according to Section 7.0.

5.6 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (if necessary), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with one or two surrogates (e.g., decafluorobiphenyl or other PAHs not expected to be present in the sample) recommended to encompass the range of the temperature program used in this method. Deuterated analogs of analytes should not be used as surrogates for HPLC analysis due to coelution problems.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1. Extracts must be stored under refrigeration and must be analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction:

7.1.1 Refer to Chapter Two for guidance on choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral pH with methylene chloride, using either Method 3510 or 3520. Solid samples are extracted using either Method 3540 or 3550. To achieve maximum sensitivity with this method, the extract must be concentrated to 1 mL.

7.1.2 Prior to HPLC analysis, the extraction solvent must be exchanged to acetonitrile. The exchange is performed during the K-D procedures listed in all of the extraction methods. The exchange is performed as follows.

7.1.2.1 Following K-D of the methylene chloride extract to 1 mL using the macro-Snyder column, allow the apparatus to cool and drain for at least 10 min.

Column: HC-ODS SIL-X
Mobile Phase: 40% to 100% Acetonitrile in Water
Dectector: Fluorescence

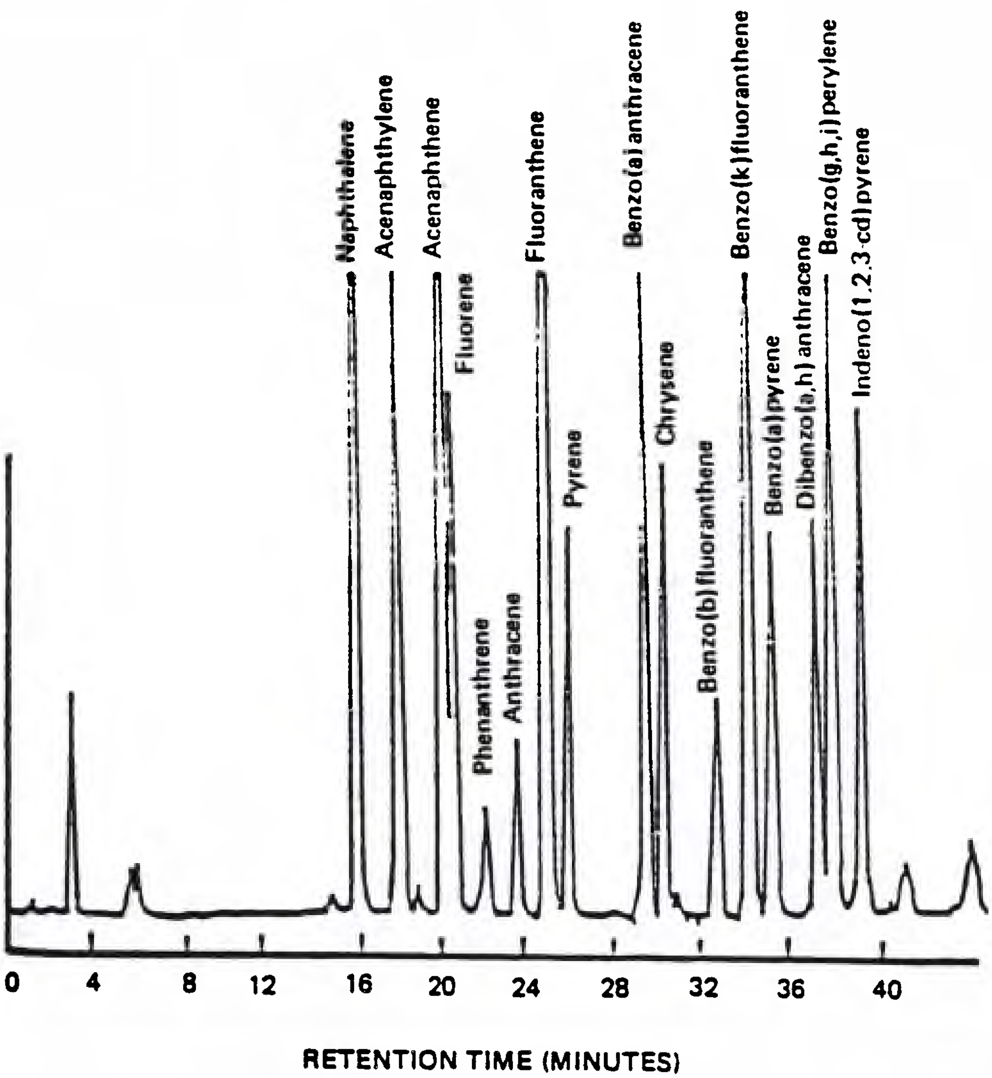


Figure 1. Liquid chromatogram of polynuclear aromatics.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in Method 8000, Section 8.10).

8.3.1 If recovery is not within limits, the following procedures are required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

9.0 METHOD PERFORMANCE

9.1 The method was tested by 16 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 0.1 to 425 ug/L. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 4.

9.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 8 x MDL to 800 x MDL with the following exception: benzo(ghi)perylene recovery at 80 x and 800 x MDL were low (35% and 45%, respectively).

9.3 The accuracy and precision obtained will be determined by the sample matrix, sample-preparation technique, and calibration procedures used.

10.0 REFERENCES

1. "Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters, Category 9 - PAHs," Report for EPA Contract 68-03-2624 (in preparation).

2. Sauter, A.D., L.D. Betowski, T.R. Smith, V.A. Strickler, R.G. Beimer, B.N. Colby, and J.E. Wilkinson, "Fused Silica Capillary Column GC/MS for the Analysis of Priority Pollutants," Journal of HRC&CC 4, 366-384, 1981.

3. "Determination of Polynuclear Aromatic Hydrocarbons in Industrial and Municipal Wastewaters," EPA-600/4-82-025, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, September 1982.

TABLE 3. QC ACCEPTANCE CRITERIA[a]

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range p, p_s (%)
Acenaphthene	100	40.3	D-105.7	D-124
Acenaphthylene	100	45.1	22.1-112.1	D-139
Anthracene	100	28.7	11.2-112.3	D-126
Benzo(a)anthracene	10	4.0	3.1-11.6	12-135
Benzo(a)pyrene	10	4.0	0.2-11.0	D-128
Benzo(b)fluoranthene	10	3.1	1.8-13.8	6-150
Benzo(ghi)perylene	10	2.3	D-10.7	D-116
Benzo(k)fluoranthene	5	2.5	D-7.0	D-159
Chrysene	10	4.2	D-17.5	D-199
Dibenzo(a,h)anthracene	10	2.0	0.3-10.0	D-110
Fluoranthene	10	3.0	2.7-11.1	14-123
Fluorene	100	43.0	D-119	D-142
Indeno(1,2,3-cd)pyrene	10	3.0	1.2-10.0	D-116
Naphthalene	100	40.7	21.5-100.0	D-122
Phenanthrene	100	37.7	8.4-133.7	D-155
Pyrene	10	3.4	1.4-12.1	D-140

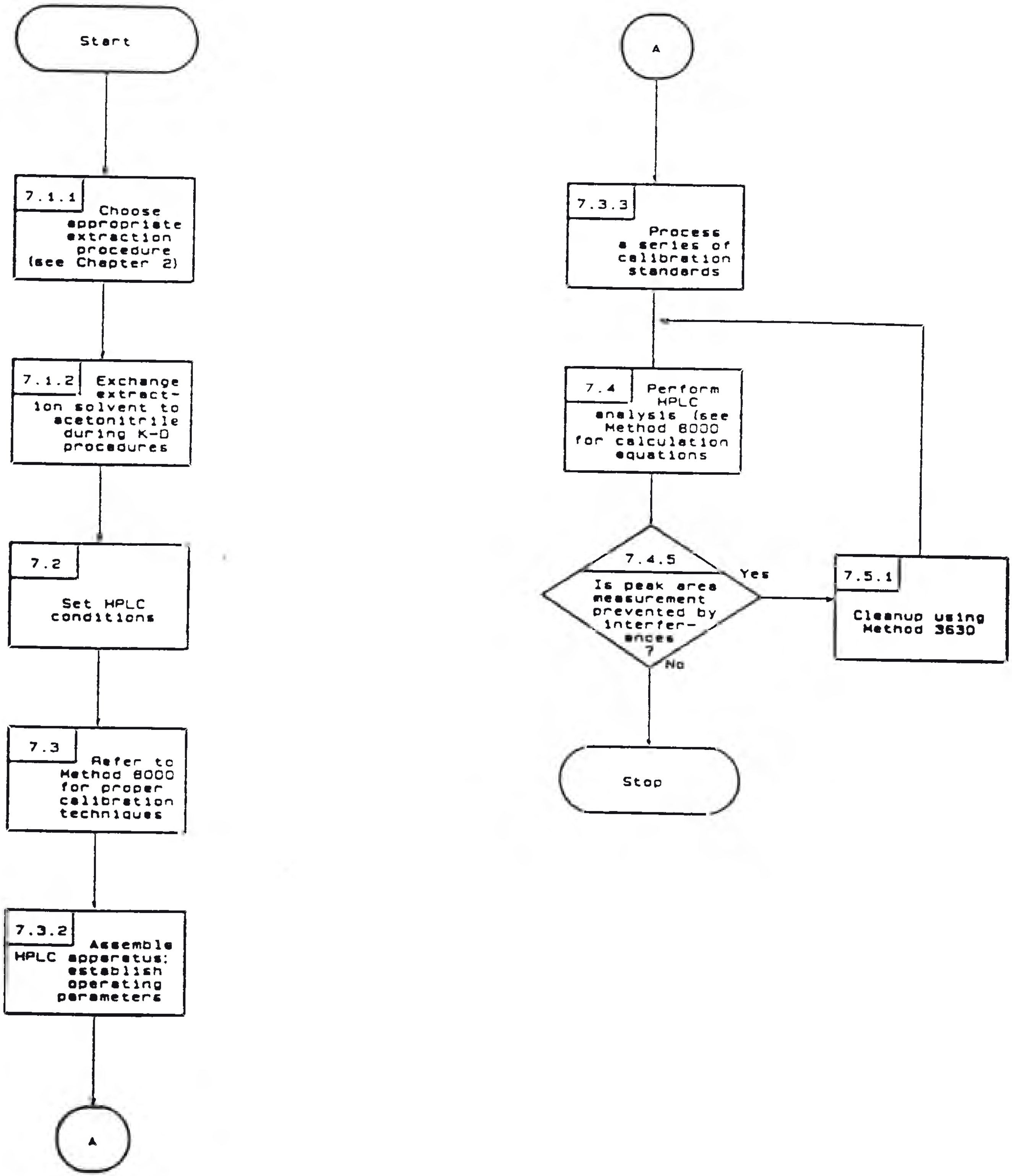
s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

p, p_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 610. These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.


Start
7.1.1
Choose appropriate extraction procedure (see Chapter 2)
7.1.2
Exchange extraction solvent to acetonitrile during K-D procedures
7.2
Set HPLC conditions
7.3
Refer to Method 8000 for proper calibration techniques
7.3.2
Assemble HPLC apparatus; establish operating parameters
A
A
7.3.3
Process a series of calibration standards
7.4
Perform HPLC analysis (see Method 8000 for calculation equations
7.4.5
Is peak area measurement prevented by interferences ?
Yes
No
7.5.1
Cleanup using Method 3630
Stop

4.4 MISCELLANEOUS SCREENING METHODS

METHOD 3520

CONTINUOUS LIQUID-LIQUID EXTRACTION

1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure for isolating organic compounds from aqueous samples. The method also describes concentration techniques suitable for preparing the extract for the appropriate determinative steps described in Step 4.3 of Chapter Four.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly soluble organics in preparation for a variety of chromatographic procedures.

1.3 Method 3520 is designed for extraction solvents with greater density than the sample. Continuous extraction devices are available for extraction solvents that are less dense than the sample. The analyst must demonstrate the effectiveness of any such automatic extraction device before employing it in sample extraction.

2.0 SUMMARY OF METHOD

2.1 A measured volume of sample, usually 1 liter, is placed into a continuous liquid-liquid extractor, adjusted, if necessary, to a specific pH (see Table 1), and extracted with organic solvent for 18-24 hours. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the determinative step being employed.

3.0 INTERFERENCES

3.1 Refer to Method 3500.

4.0 APPARATUS AND MATERIALS

4.1 Continuous liquid-liquid extractor - Equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf Extractor -- Ace Glass Company, Vineland, New Jersey, P/N 6841-10, or equivalent).

4.2 Drying column - 20 mm i.d. Pyrex chromatographic column with Pyrex glass wool at bottom and a Teflon stopcock.

<u>NOTE</u>: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

4.3 Kuderna-Danish (K-D) apparatus

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Step 4.1.

7.0 PROCEDURE

7.1 Using a graduated cylinder, measure out 1 liter (nominal) of sample and transfer it to the continuous extractor. If high concentrations are anticipated, a smaller volume may be used and then diluted with water to 1 liter. Check the pH of the sample with wide-range pH paper and adjust the pH, if necessary, to the pH indicated in Table 1. Pipet 1.0 mL of the surrogate standard spiking solution into each sample into the extractor and mix well. (See Method 3500 for details on the surrogate standard solution and the matrix spike solution.) For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount of the surrogates and matrix spiking compounds added to the sample should result in a final concentration of 100 ng/uL of each base/neutral analyte and 200 ng/uL of each acid analyte in the extract to be analyzed (assuming a 1 uL injection). If Method 3640, Gel-Permeation Cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column.

7.2 Add 300-500 mL of methylene chloride to the distilling flask. Add several boiling chips to the flask.

7.3 Add sufficient water to the extractor to ensure proper operation and extract for 18-24 hours.

7.4 Allow to cool; then detach the boiling flask. If extraction at a secondary pH is not required (see Table 1), the extract is dried and concentrated as described in Steps 7.7 through 7.11.

7.5 Carefully, while stirring, adjust the pH of the aqueous phase to < 2 with sulfuric acid (1:1). Attach a clean distilling flask containing 500 mL of methylene chloride to the continuous extractor. Extract for 18-24 hours, allow to cool, and detach the distilling flask.

7.6 If performing GC/MS analysis (Method 8250 or 8270), the acid and base/neutral extracts may be combined prior to concentration. However, in some situations, separate concentration and analysis of the acid and base/neutral extracts may be preferable (e.g. if for regulatory purposes the presence or absence of specific acid or base/neutral compounds at low concentrations must be determined, separate extract analyses may be warranted).

7.7 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask.

7.8 Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the flask which contained the solvent extract with

8.0 QUALITY CONTROL

8.1 Any reagent blanks, matrix spike, or replicate samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.2 Refer to Chapter One for specific quality control procedures and Method 3500 for extraction and sample-preparation procedures.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

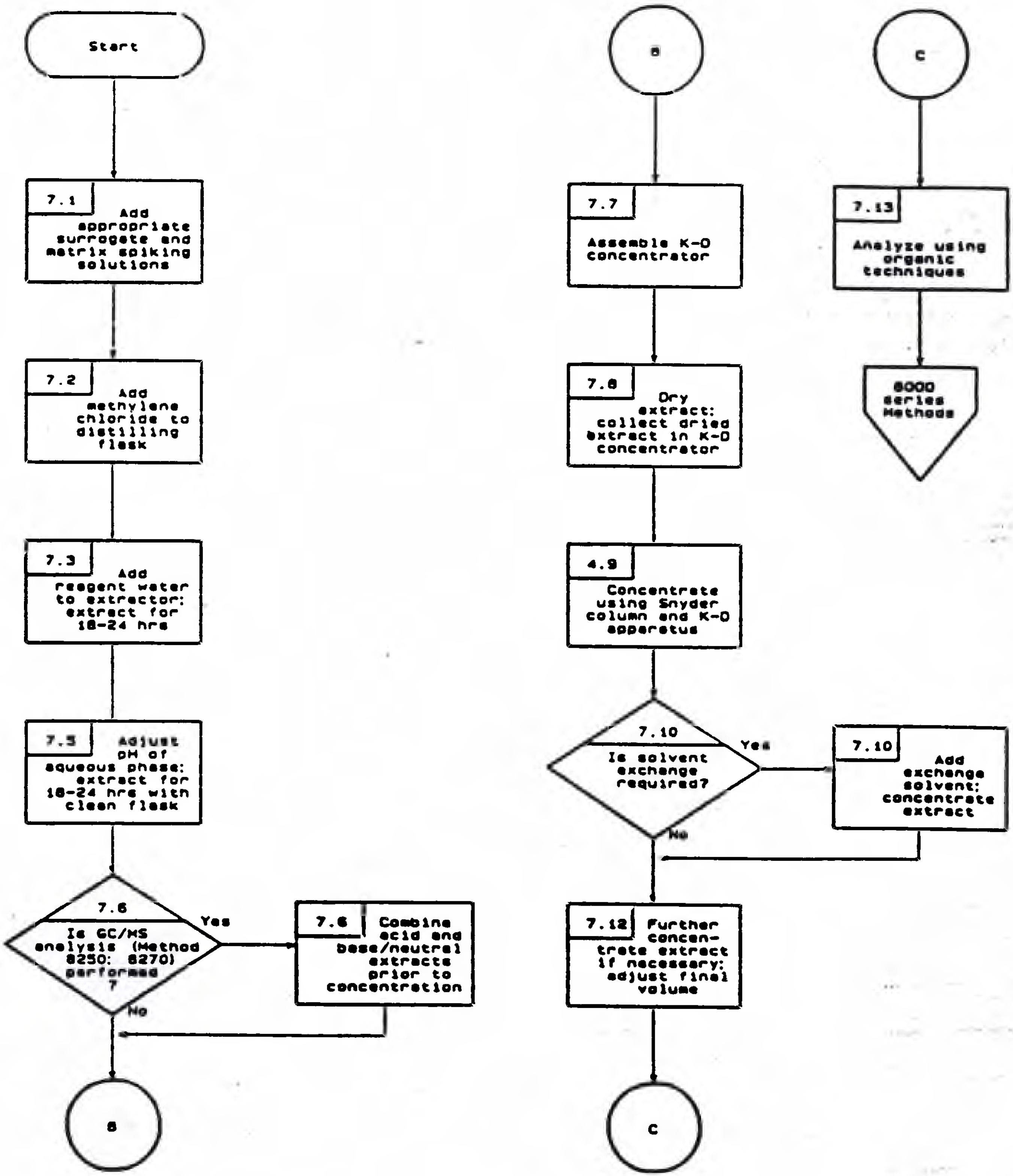
10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

2. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

3. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

METHOD 3520
CONTINUOUS LIQUID-LIQUID EXTRACTION



METHOD 3630

SILICA GEL CLEANUP

1.0 SCOPE AND APPLICATION

1.1 Silica gel is a regenerative adsorbent of amorphous silica with weakly acidic properties. It is produced from sodium silicate and sulfuric acid. Silica gel can be used for column chromatography and is for separating the analytes from interfering compounds of a different chemical polarity.

1.2 General applications (Gordon and Ford):

1.2.1 **Activated:** Heated at 150-160°C for several hours. USES: Separation of hydrocarbons.

1.2.2 **Deactivated:** Containing 10-20% water. USES: An adsorbent for most functionalities with ionic or nonionic characteristics, including alkaloids, sugar esters, glycosides, dyes, alkali metal cations, lipids, glycerides, steroids, terpenoids and plasticizers. The disadvantages of deactivated silica gel are that the solvents methanol and ethanol decrease adsorbent activity.

1.3 Specific applications: This method includes guidance for cleanup of sample extracts containing polynuclear aromatic hydrocarbons and derivatized phenolic compounds.

2.0 SUMMARY OF METHOD

2.1 The column is packed with the required amount of adsorbent, topped with a water adsorbent, and then loaded with the sample to be analyzed. Elution of the analytes is effected with a suitable solvent(s) leaving the interfering compounds on the column. The eluate is then concentrated.

3.0 INTERFERENCES

3.1 A reagent blank should be performed for the compounds of interest prior to the use of this method. The level of interferences must be below the method detection limit before this method is performed on actual samples.

3.2 More extensive procedures than those outlined in this method may be necessary for reagent purification.

4.0 APPARATUS AND MATERIALS

4.1 Chromatographic column: 250-mm long x 10-mm I.D.; with Pyrex glass wool at bottom and a Teflon stopcock.
NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits

7.0 PROCEDURE

7.1 Polynuclear aromatic hydrocarbons:

7.1.1 Before the silica gel cleanup technique can be utilized, the extract solvent must be exchanged to cyclohexane. Add 1 to 10 mL of the sample extract (in methylene chloride) and a boiling chip to a clean K-D concentrator tube. Add 4 mL of cyclohexane and attach a two-ball micro-Snyder column. Prewet the column by adding 0.5 mL of methylene chloride to the top. Place the micro-K-D apparatus on a boiling (100°C) water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of the liquid reaches 0.5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of cyclohexane. Adjust the extract volume to about 2 mL.

7.1.2 Prepare a slurry of 10 g of activated silica gel in methylene chloride and place this into a 10-mm I.D. chromatographic column. Tap the column to settle the silica gel and elute the methylene chloride. Add 1 to 2 cm of anhydrous sodium sulfate to the top of the silica gel.

7.1.3 Preelute the column with 40 mL of pentane. The rate for all elutions should be about 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, transfer the 2 mL cyclohexane sample extract onto the column using an additional 2 mL cyclohexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 25 mL of pentane and continue the elution of the column. Discard this pentane eluate.

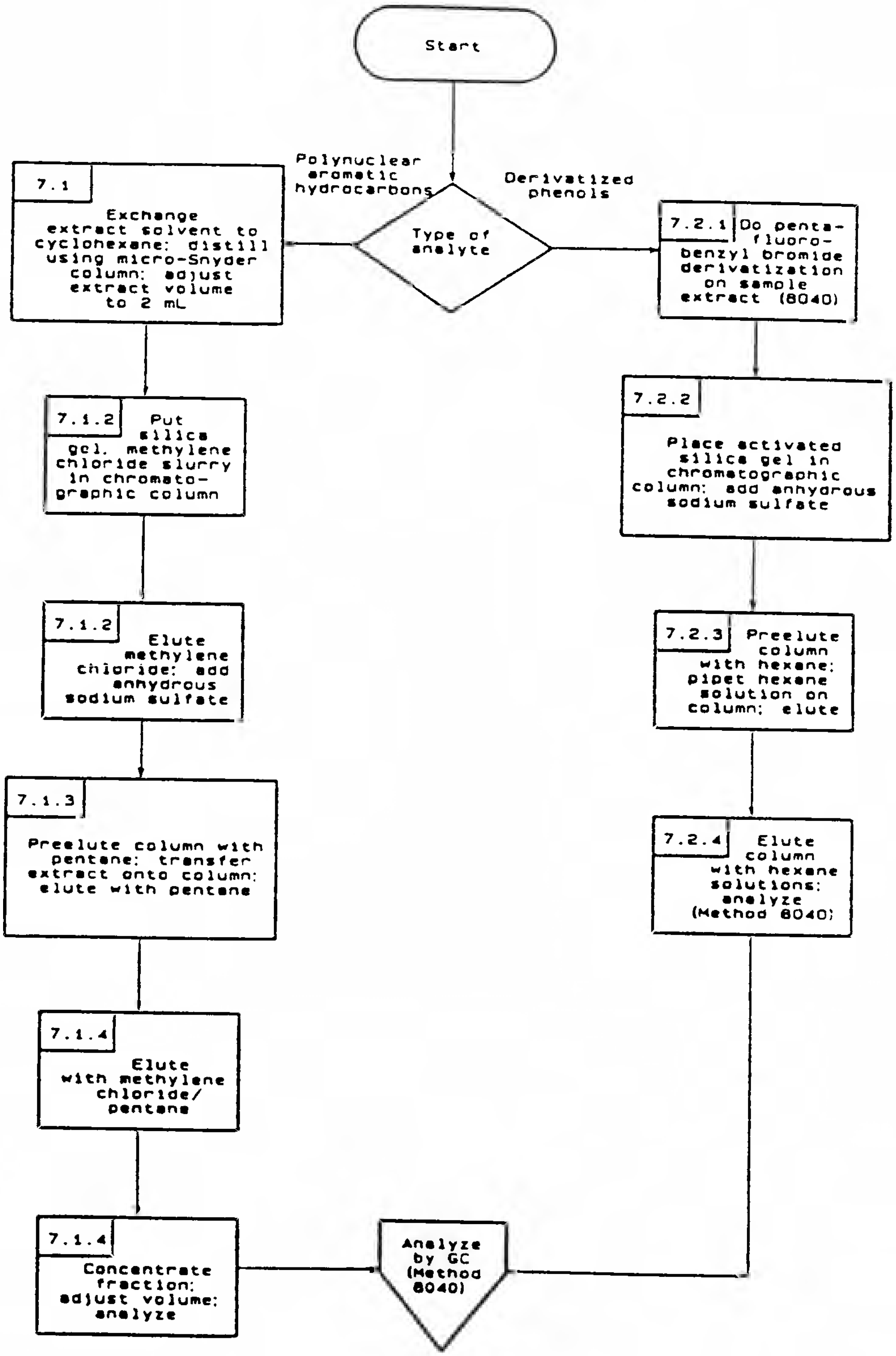
7.1.4 Next, elute the column with 25 mL of methylene chloride/-pentane (2:3)(v/v) into a 500-mL K-D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction to whatever volume is required (1-10 mL). Proceed with HPLC or GC analysis. Components that elute in this fraction are:

Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(ghi)perylene
Benzo(k)fluoranthene
Chrysene
Dibenzo(a,h)anthracene
Fluoranthene
Fluorene

10.0 REFERENCES

1. Gordon, A.J., and R.A. Ford, The Chemist's Companion: A Handbook of Practical Data, Techniques, and References, (New York: John Wiley & Sons, Inc.), pp. 372, 374, and 375, 1972.

2. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.


Start
Polynuclear aromatic hydrocarbons
Derivatized phenols
Type of analyte
7.1 Exchange extract solvent to cyclohexane: distill using micro-Snyder column: adjust extract volume to 2 mL
7.1.2 Put silica gel. methylene chloride slurry in chromato-graphic column
7.1.2 Elute methylene chloride: add anhydrous sodium sulfate
7.1.3 Preelute column with pentane: transfer extract onto column: elute with pentane
7.1.4 Elute with methylene chloride/ pentane
7.1.4 Concentrate fraction: adjust volume: analyze
7.2.1 Do penta-fluoro-benzyl bromide derivatization on sample extract (8040)
7.2.2 Place activated silica gel in chromatographic column: add anhydrous sodium sulfate
7.2.3 Preelute column with hexane: pipet hexane solution on column: elute
7.2.4 Elute column with hexane solutions: analyze (Method 8040)
Analyze by GC (Method 8040)

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request

[X] Regional Transmittal   [ ] Telephone Request

A. EPA Region/Client: Region 10

B. Authorized By: Bruce Woods (206) 553-1193

C. Prepared By: Laura Castrilli (206) 553-4323

D. Date of Request: March 8, 1991

E. Site Name: Ridgefield Brick and Tile (RBT)
Ridgefield, Washington

F. 2 digit Superfund site identifier: N/A

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

   The requested analytical service will be used to determine the concentration of chlorophenols in ground water (System No. 1) and leachate (System No. 2) at RBT.

2. Definition <u>and</u> number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):

   This will be a split sampling event.

   Six aqueous environmental samples, two aqueous duplicate samples, and two aqueous equipment rinsate blanks (**10 samples/blanks total**) will be received to analyze for chlorophenols. From previous sampling data, these samples and blanks are expected to contain low concentrations of chlorophenols.

3. **Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):**

   The purpose of this analysis will be to supply supporting analytical data to EPA to aid in characterizing the RBT site. EPA is considering a clean closure option for the RBT landfill.

4. **Estimated date(s) of collection:** March 27 and 28, 1991

5. **Estimated date(s) and method of shipment:**

   Samples will be shipped by Federal Express on March 27 and 28, 1991 following each day's sampling activities.

6. **Number of days analysis and data required after laboratory receipt of samples:**

   Samples must be preserved with 1.5 mL of 10% $Na_2S_2O_3$ per liter and stored in the dark at 4°C. The laboratory must extract these samples within five days of collection and analysis must follow within 40 days.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

   The protocol to be used for chlorophenol analysis is a modified version of Method 8040 from Test Methods for Evaluating Solid Waste (SW-846). Copies of both Method 8040 and its modified version are attached to this SAS request.

8. **Special technical instructions (if outside protocol requirements, specify compound names, SAS numbers, detection limits, etc.):**

   Special instructions are included in the modified version of Method 8040. Chlorophenols may be detected as low as 1.0 μg/L using this modified version.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.**

   Data will be reported in the standard Contract Laboratory program Routine Analytical Service format.

11. **Name of sampling/shipping contact:**

Gary Bruno
PRC Environmental Management, Inc.

Phone: 206/624-2692

12. **Data Requirements**

Parameter	Quantitation Limit	Precision (percent or Concentration)
Chlorophenols	1.0 µg/L	± 20

13. **QC Requirements**

Audits required	Frequency of Audits	Limits (percent or Concentration)
Laboratory Control Samples	Once per 20 Samples or once per 12 hours of continuous assay.	± 10 %
Analytical System Preparation	See Method 8040 Sections 5.5 and 7.3 and Method 8000 Sections 7.4.2 and 7.5. (Copies of Methods 8040 and 8000 are attached to this SAS request.	See Method 8040 Table 4.
MS/MSD	Once per 20 samples	± 30 %
Laboratory Duplicates	Once per 20 samples	± 20 %

14. **Action Required if Limits are Exceeded**

Recalibrate analysis instrument as indicated and reassay or call Bruce Woods, QA chemist, at 206-553-1193 (FTS 399-1193) or Gerald Muth, CLP DPO, at 206-871-0748 (FTS 390-1282), immediately, for problem resolution.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

METHOD 8040

PHENOLS

1.0 SCOPE AND APPLICATION

1.1 Method 8040 is used to determine the concentration of various phenolic compounds. Table 1 indicates compounds that may be analyzed by this method and lists the method detection limit for each compound in water. Table 2 lists the practical quantitation limit (PQL) for all matrices.

2.0 SUMMARY OF METHOD

2.1 Method 8040 provides gas chromatographic conditions for the detection of phenolic compounds. Prior to analysis, samples must be extracted using appropriate techniques (see Chapter Two for guidance). Both neat and diluted organic liquids (Method 3580, Waste Dilution) may be analyzed by direct injection. A 2- to 5-uL sample is injected into a gas chromatograph using the solvent flush technique, and compounds in the GC effluent are detected by a flame ionization detector (FID).

2.2 Method 8040 also provides for the preparation of pentafluorobenzyl-bromide (PFB) derivatives, with additional cleanup procedures for electron capture gas chromatography. This is to reduce detection limits of some phenols and to aid the analyst in the elimination of interferences.

3.0 INTERFERENCES

3.1 Refer to Methods 3500, 3600, and 8000.

3.2 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by analyzing calibration and reagent blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.3 Interferences coextracted from samples will vary considerably from source to source, depending upon the waste being sampled. Although general cleanup techniques are recommended as part of this method, unique samples may require additional cleanup.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 Gas Chromatograph - Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak areas and/or peak heights is recommended.

5.2 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified.

5.3 Hexane, $CH_3(CH_2)_4CH_3$. Pesticide quality or equivalent.

5.4 2-Propanol, $(CH_3)_2CHOH$. Pesticide quality or equivalent.

5.5 Toluene, $C_6H_5CH_3$. Pesticide quality or equivalent.

5.6 Derivatization reagent - Add 1 mL pentafluorobenzyl bromide and 1 g 18-crown-6-ether to a 50-mL volumetric flask and dilute to volume with 2-propanol. Prepare fresh weekly. This operation should be carried out in a hood. Store at 4°C and protect from light.

5.6.1 Pentafluorobenzyl bromide (alpha-Bromopentafluorotoluene), $C_6F_5CH_2Br$. 97% minimum purity.

<u>NOTE</u>: This chemical is a lachrymator.

5.6.2 18-crown-6-ether (1,4,7,10,13,16-Hexaoxacyclooctadecane) - 98% minimum purity.

<u>NOTE</u>: This chemical is highly toxic.

5.7 Potassium carbonate (Powdered), K_2CO_3.

5.8 Stock standard solutions

5.8.1 Prepare stock standard solution at a concentration of 1.00 ug/uL by dissolving 0.0100 g of assayed reference material in 2-propanol and diluting to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

5.8.2 Transfer the stock standard solutions into bottles with Teflon lined screw-caps. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.8.3 Stock standard solutions must be replaced after one year, or sooner if comparison with check standards indicates a problem.

5.9 Calibration standards - Calibration standards at a minimum of five concentration levels should be prepared through dilution of the stock standards with 2-propanol. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC.

7.1.2.2 Increase the temperature of the hot water bath to 95-100°C. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of 2-propanol. A 5-mL syringe is recommended for this operation. Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column. Prewet the column by adding about 0.5 mL of 2-propanol to the top. Place the K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration in 5-10 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 2.5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. Add an additional 2 mL of 2-propanol, add one or two clean boiling chips to the concentrator tube, and resume concentrating as before. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

7.1.2.3 Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of 2-propanol. Adjust the extract volume to 1.0 mL. Stopper the concentrator tube and store refrigerated at 4°C if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a vial with a Teflon lined screw-cap. If the extract requires no further derivatization or cleanup, proceed with gas chromatographic analysis.

7.2 Gas chromatography conditions (Recommended)

7.2.1 Column for underivatized phenols - Set nitrogen gas flow at 30 mL/min flow rate. Set column temperature at 80°C and immediately program an 8°C/min temperature rise to 150°C; hold until all compounds have eluted.

7.2.2 Column for derivatized phenols - Set 5% methane/95% argon gas flow at 30 mL/min flow rate. Set column temperature at 200°C isothermal.

7.3 Calibration - Refer to Method 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.3.1 The procedure for internal or external calibration may be used for the underivatized phenols. Refer to Method 8000 for a description of each of these procedures. If derivatization of the phenols is required, the method of external calibration should be used by injecting five or more levels of calibration standards that have also undergone derivatization and cleanup prior to instrument calibration.

7.4 Gas chromatographic analysis

where:

A = Mass of underivatized phenol represented by area of peak in sample chromatogram, determined from calibration curve (see Method 8000 Step 7.4.2), ng.

V_t = Total amount of column eluate or combined fractions from which V_i was taken, uL.

B = Total volume of hexane added in Step 7.5.5, mL.

D = Total volume of 2-propanol extract prior to derivatization, mL.

V_i = Volume injected, uL.

X = Volume of water extracted, mL, or weight of nonaqueous sample extracted, g, from Step 7.1. Either the dry or wet weight of the nonaqueous sample may be used, depending upon the specific application of the data.

C = Volume of hexane sample solution added to cleanup column (Method 3630, Step 7.2), mL.

E = Volume of 2-propanol extract carried through derivatization in Step 7.5.1, mL.

7.5 Derivatization - If interferences prevent measurement of peak area during analysis of the extract by flame ionization gas chromatography, the phenols must be derivatized and analyzed by electron capture gas chromatography.

7.5.1 Pipet a 1.0-mL aliquot of the 2-propanol stock standard solution or of the sample extract into a glass reaction vial. Add 1.0 mL derivatization reagent (Step 5.3). This amount of reagent is sufficient to derivatize a solution whose total phenolic content does not exceed 0.3 mg/mL.

7.5.2 Add approximately 3 mg of potassium carbonate to the solution and shake gently.

7.5.3 Cap the mixture and heat it for 4 hours at 80°C in a hot water bath.

7.5.4 Remove the solution from the hot water bath and allow it to cool.

7.5.5 Add 10 mL hexane to the reaction vial and shake vigorously for 1 minute. Add 3.0 mL water to the reaction vial and shake for 2 minutes.

7.5.6 Decant the organic layer into a concentrator tube and cap with a glass stopper. Proceed with cleanup procedure.

matrix. Linear equations to describe these relationships for a flame ionization detector are presented in Table 5.

9.2 The accuracy and precision obtained will be affected by the sample matrix, sample-preparation technique, and calibration procedures used.

10.0 REFERENCES

1. Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters. Category 3 - Chlorinated Hydrocarbons and Category 8 - Phenols. Report for EPA Contract 68-03-2625 (in preparation).

2. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

3. "Determination of Phenols in Industrial and Municipal Wastewaters," Report for EPA Contract 68-03-2625 (in preparation).

4. "EPA Method Validation Study Test Method 604 (Phenols)," Report for EPA Contract 68-03-2625 (in preparation).

5. Kawarahara, F.K. "Microdetermination of Derivatives of Phenols and Mercaptans by Means of Electron Capture Gas Chromatography," Analytical Chemistry, 40, 1009, 1968.

6. Provost, L.P. and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, pp. 58-63, 1983.

7. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," Journal of the Association of Official Analytical Chemists, 48, 1037, 1965.

8. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

10. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

TABLE 3.
ELECTRON CAPTURE GAS CHROMATOGRAPHY OF PFB DERIVATIVES

Parent compound	Retention time (min)	Method detection limit (ug/L)
4-Chloro-2-methylphenol	4.8	1.8
2-Chlorophenol	3.3	0.58
2,4-Dichlorophenol	5.8	0.68
2,4-Dimethylphenol	2.9	0.63
2,4-Dinitrophenol	46.9	
2-Methyl-4,6-dinitrophenol	36.6	
2-Nitrophenol	9.1	0.77
4-Nitrophenol	14.0	0.70
Pentachlorophenol	28.8	0.59
Phenol	1.8	2.2
2,4,6-Trichlorophenol	7.0	0.58

TABLE 5.
METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Parameter	Accuracy, as recovery, x′ (ug/L)	Single analyst precision, s_r' (ug/L)	Overall precision, S′ (ug/L)
4-Chloro-3-methylphenol	0.87C-1.97	0.11x-0.21	0.16x+1.41
2-Chlorophenol	0.83C-0.84	0.18x+0.20	0.21x+0.75
2,4-Dichlorophenol	0.81C+0.48	0.17x-0.02	0.18x+0.62
2,4-Dimethylphenol	0.62C-1.64	0.30x-0.89	0.25x+0.48
4,6-Dinitro-2-methylphenol	0.84C-1.01	0.15x+1.25	0.19x+5.85
2,4-Dinitrophenol	0.80C-1.58	0.27x-1.15	0.29x+4.51
2-Nitrophenol	0.81C-0.76	0.15x+0.44	0.14x+3.84
4-Nitrophenol	0.46C+0.18	0.17x+2.43	0.19x+4.79
Pentachlorophenol	0.83C+2.07	0.22x-0.58	0.23x+0.57
Phenol	0.43C+0.11	0.20x-0.88	0.17x+0.77
2,4,6-Trichlorophenol	0.86C-0.40	0.10x+0.53	0.13x+2.40

x′ = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of x, in ug/L.

S′ = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in ug/L.

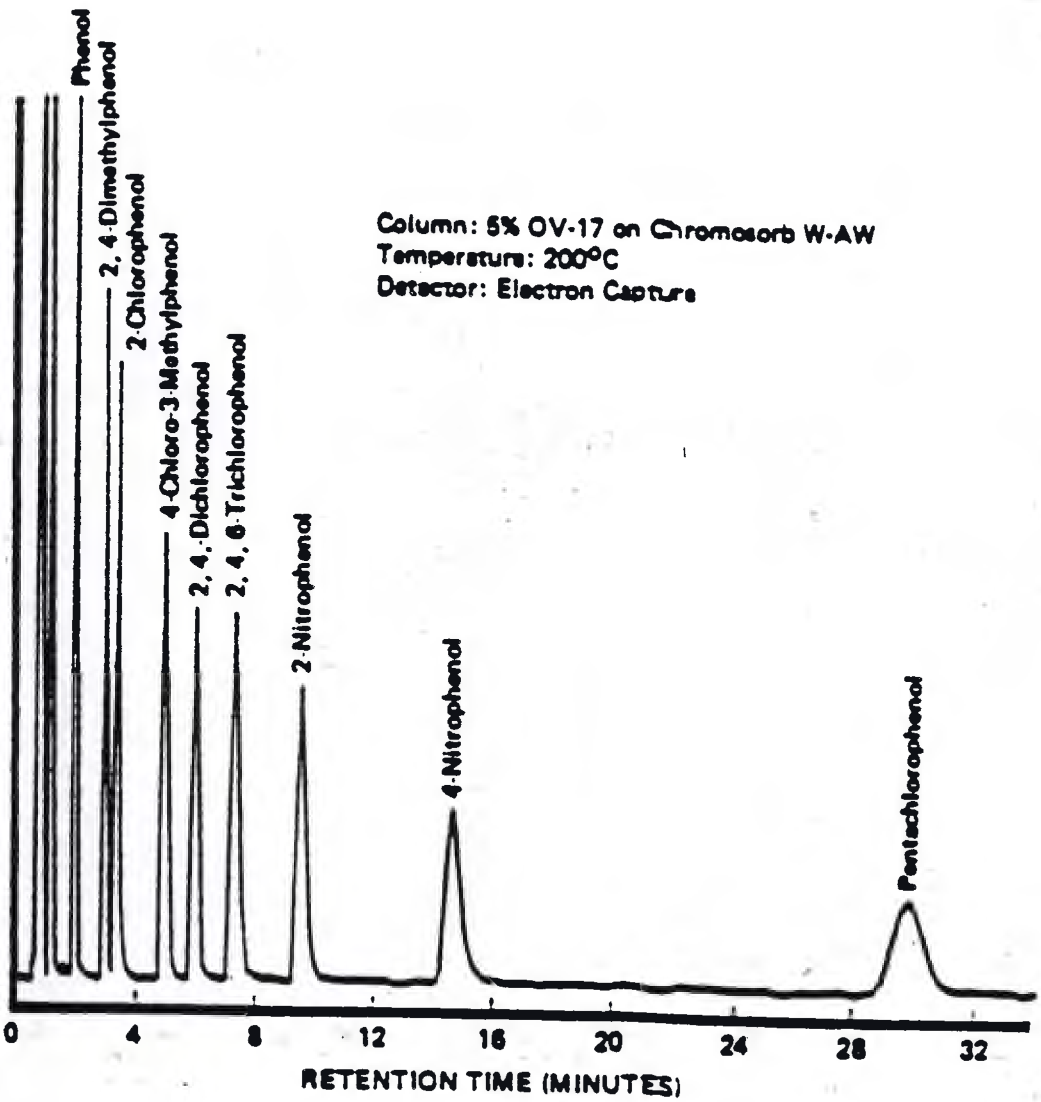
C = True value for the concentration, in ug/L.

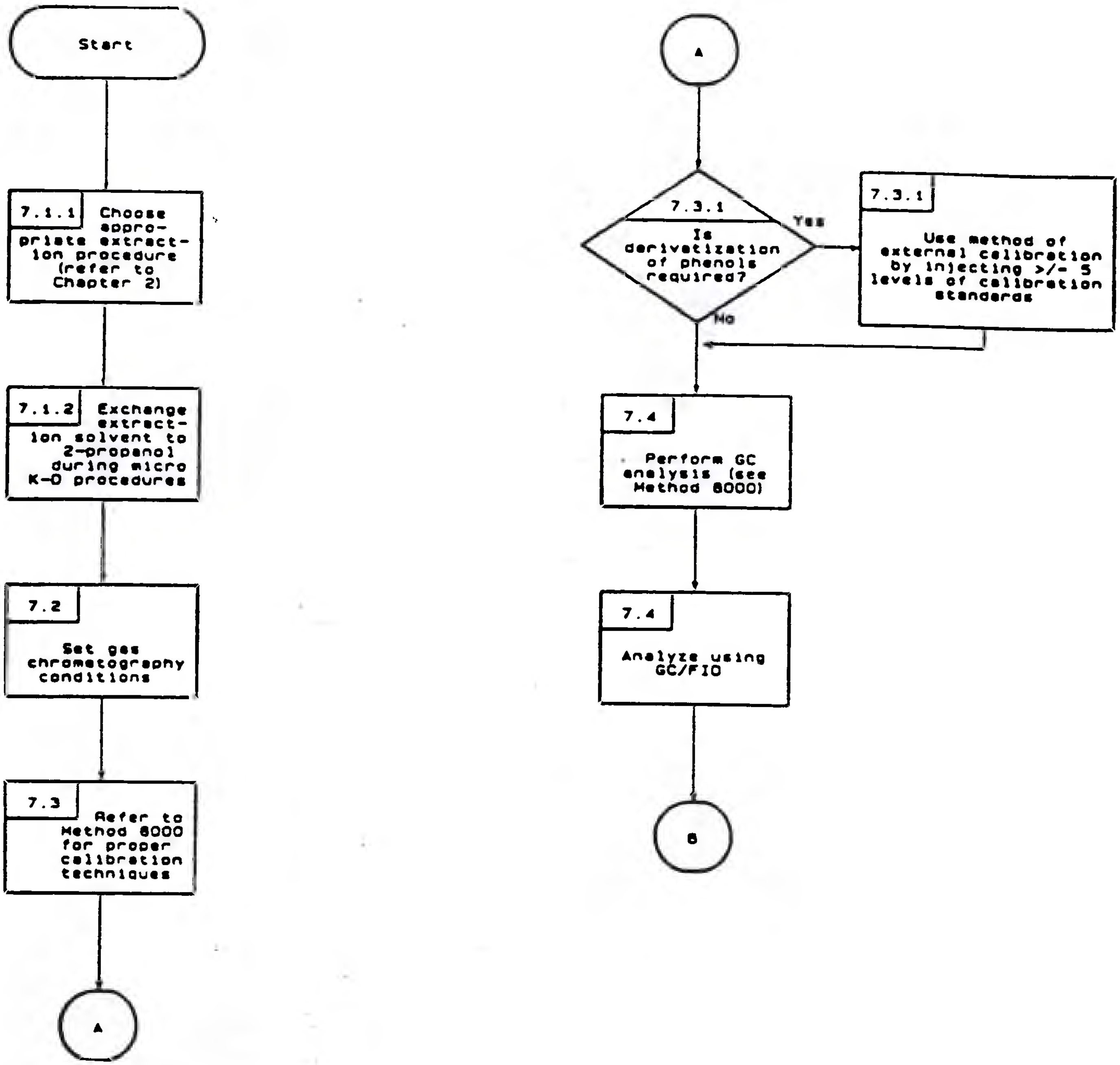
x = Average recovery found for measurements of samples containing a concentration of C, in ug/L.

[a]From 40 CFR Part 136 for Method 604.

Figure 2.

Gas chromatogram of PFB derivatives of phenols.




Start
7.1.1 Choose appro-priate extract-ion procedure (refer to Chapter 2)
7.1.2 Exchange extract-ion solvent to 2-propanol during micro K-D procedures
7.2 Set gas chromatography conditions
7.3 Refer to Method 8000 for proper calibration techniques
A
A
7.3.1 Is derivatization of phenols required?
Yes
No
7.3.1 Use method of external calibration by injecting >/= 5 levels of calibration standards
7.4 Perform GC analysis (see Method 8000)
7.4 Analyze using GC/FID
B

Analysis of Phenols in Water and Soil by Modified SW846 Method 8040:

Analytes:

Sample Matrices: Low concentration water and soil samples (specify whether soil, surface water, groundwater, drinking water, waste or leachate).

Analytical Procedure and Quantitation Limits:

1. Water Sample Extraction: A 20 mL sample aliquot is rinsed with about 20 mL of diethyl ether. The sample is acidified with 1.0 mL of conc. hydrochloric acid (check that pH is <4) and extracted with 10 mL of diethyl ether. One-half of the extract (equal to 10 mL of the original sample) is concentrated under nitrogen to about 1 mL, treated with ethereal diazomethane, and adjusted to 5.0 mL with isooctane.
2. Soil or Sediment Sample Extraction: A 10 g subsample is extracted by shaking for 2 hours with 20 mL of diethyl ether containing 1% (v/v) sulfuric acid. After allowing the particles to settle, a 1.0 mL aliquot (equal to 0.5 g of the original sample) is rinsed with 5 mL of water, concentrated under nitrogen to about 1 mL, treated with diazomethane and adjusted to 5.0 mL with isooctane.
3. Diazomethane is a carcinogen and can explode under certain conditions. Refer to Section 7.3.1 of Method 8150 for precautions.
4. Follow SW846 Method 8040 for the GC analysis of water and soil extracts. Use the methods described above for sample extraction to obtain lower detection limits.
5. The contract required quantitation limits (CRQL) are 1.0 ug/L for water samples and 10 ug/Kg for soil samples.
6. Capillary columns may be used for this analysis, as long as the laboratory demonstrates that the analysis meets all the performance and QA/QC criteria specified in Method 8040 and in this contract.

Contract Holding Times: Contract required holding times are five (5) days for the extraction of water samples, ten (10) days for the extraction of soil samples and forty (40) days for the analysis of extracts, from the date of sample receipt by the laboratory.

Calibration Procedure and Criteria: Calibrate according to Section 7.3 of Method 8040, and Sections 7.4.2 and 7.5 of Method 8000, with the following specifications.

1. All standards should be prepared with phenols as the methyl esters.
2. Five-point initial calibration curve with low standards at concentrations of the CRQL or lower is required.

Phenols in Water and Soil by Modified SW846 Method 8040 (Continued):

3. A continuing calibration at the mid-point concentration for each analyte is to be analyzed at the beginning of each day and after each group of 10 samples.
4. Less than 10% relative standard deviation (%RSD) in calibration factors (CF) for initial calibration standards and less than ±15% difference (%D) in CF for daily calibrations are required.

Internal Quality Control Checks, Control Limits and Corrective Actions:

1. Analyze method blanks at a frequency of one per group of 20 or fewer samples. The method blanks must be free of phenols and any interfering peaks at or above the detection limits.
2. A phenolic surrogate (2-fluorophenol or 2,4,6-tribromophenol is recommended) must be spiked into the standards, samples, method blanks and QC samples (see Sections 5.7 and 8.3 of Method 8040). The amount of surrogate added must be at least 10 times the instrument detection limit. Recoveries of 50-125% are required.
3. Second column confirmation is required for all positive results reported.
4. Sample extracts containing one or more analytes at concentrations above the initial calibration range are to be diluted and re-analyzed. Report the results and documentation for both analyses.
5. Analyze matrix spikes and matrix spike duplicates (MS/MSD) at the frequency of one per group of 20 or fewer samples. Concentration of the matrix spike solution should be such that the final extracts contain amounts at the mid range of the calibration curve. Recoveries of 60-125% are required.
6. If above control limits are exceeded, take appropriate actions to correct the problems and re-analyze the affected samples.

Data Calculations and Reporting Units:

1. Calculate the CF and the concentration of individual analytes using the equations in Sections 7.4.2 and 7.8.1 of Method 8000. The sample results are to be reported in the concentration units of micrograms per liter (ug/L) for water samples and micrograms per kilogram (ug/Kg) on a dry weight basis for soil samples.
2. All records of analysis, dilution and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example of the calculations in the data package.

Phenols in Water and Soil by Modified SW846 Method 8040 (continued):

Documentation and Deliverables.

1. Report documentation for sample and blank results, date and time of analysis, surrogate recovery, MS/MSD recovery, blanks summary, analytical sequence, initial and continuing calibrations (including retention time windows), and compound identification. Use forms equivalent to the CLP Forms I, II, III, IV, VI, IX and X.
2. Provide all raw data, including chromatograms and area printouts/ quantitation reports.
3. Raw data and summary forms are to be organized systematically and each page is to be numbered.

METHOD 8000

GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 Gas chromatography is a quantitative analytical technique useful for organic compounds capable of being volatilized without being decomposed or chemically rearranged. Gas chromatography (GC), also known as vapor phase chromatography (VPC), has two subcategories distinguished by: gas-solid chromatography (GSC), and gas-liquid chromatography (GLC) or gas-liquid partition chromatography (GLPC). This last group is the most commonly used, distinguished by type of column adsorbent or packing.

1.2 The gas chromatographic methods are recommended for use only by, or under the close supervision of, experienced residue analysts.

2.0 SUMMARY OF METHOD

2.1 Each organic analytical method that follows provides a recommended technique for extraction, cleanup, and occasionally, derivatization of the samples to be analyzed. Before the prepared sample is introduced into the GC, a procedure for standardization must be followed to determine the recovery and the limits of detection for the analytes of interest. Following sample introduction into the GC, analysis proceeds with a comparison of sample values with standard values. Quantitative analysis is achieved through integration of peak area or measurement of peak height.

3.0 INTERFERENCES

3.1 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device must be rinsed out between samples with water or solvent. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank or of water to check for cross contamination. For volatile samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the syringe or purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph - Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak height and/or peak areas is recommended.

4.2 Gas chromatographic columns - See the specific determinative method. Other packed or capillary (open-tubular) columns may be used if the requirements of Step 8.6 are met.

$$\text{Calibration factor} = \frac{\text{Total Area of Peak}^{*}}{\text{Mass injected (in nanograms)}}$$

*For multiresponse pesticides/PCBs use the total area of all peaks used for quantitation.

7.4.2.3 The working calibration curve or calibration factor must be verified on each working day by the injection of one or more calibration standards. The frequency of verification is dependent on the detector. Detectors, such as the electron capture detector, that operate in the sub-nanogram range are more susceptible to changes in detector response caused by GC column and sample effects. Therefore, more frequent verification of calibration is necessary. The flame ionization detector is much less sensitive and requires less frequent verification. If the response for any analyte varies from the predicted response by more than $\pm$ 15%, a new calibration curve must be prepared for that analyte.

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_1} \times 100$$

where:

R_1 = Calibration Factor from first analysis.

R_2 = Calibration Factor from succeeding analyses.

7.4.3 Internal standard calibration procedure

7.4.3.1 To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Due to these limitations, no internal standard applicable to all samples can be suggested.

7.4.3.2 Prepare calibration standards at a minimum of five concentration levels for each analyte of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards and dilute to volume with an appropriate solvent. One of the standards should be at a concentration near, but above, the method detection limit. The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.4.3.3 Inject each calibration standard using the same introduction technique that will be applied to the actual samples (e.g. 2- to 5-uL injection, purge-and-trap, etc.). Tabulate the peak height or area responses against the concentration of each compound and internal standard. Calculate response factors (RF) for each compound as follows:

7.5.2.2 In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

7.5.3 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

7.6 Gas chromatographic analysis

7.6.1 Introduction of organic compounds into the gas chromatograph varies depending on the volatility of the compound. Volatile organics are primarily introduced by purge-and-trap (Method 5030). However, there are limited applications where direct injection is acceptable. Use of Method 3810 or 3820 as a screening technique for volatile organic analysis may be valuable with some sample matrices to prevent overloading and contamination of the GC systems. Semivolatile organics are introduced by direct injection.

7.6.2 The appropriate detector(s) is given in the specific method.

7.6.3 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with multilevel calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

7.6.4 Direct Injection - Inject 2-5 uL of the sample extract using the solvent flush technique. Smaller (1.0-uL) volumes can be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 uL and the resulting peak size in area units or peak height.

7.6.5 If the responses exceed the linear range of the system, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

7.6.6 If peak detection is prevented by the presence of interferences, further cleanup is required.

7.6.7 Examples of chromatograms for the compounds of interest are frequently available in the referring analytical method.

7.6.8 Calibrate the system immediately prior to conducting any analyses (see Step 7.4). A midlevel standard must also be injected at intervals specified in the method and at the end of the analysis sequence. The calibration factor for each analyte to be quantitated, must not exceed a 15% difference when compared to the initial standard of injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

7.7.3.1 Place a beaker beneath the injector port inside the GC oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone and then toluene; catching the rinsate in the beaker.

7.7.3.2 Prepare a solution of deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, serially rinse the injector body with toluene, methanol,acetone, and hexane. Reassemble the injector and replace the GC column.

7.8 Calculations

7.8.1 External standard calibration - The concentration of each analyte in the sample may be determined by calculating the amount of standard purged or injected, from the peak response, using the calibration curve or the calibration factor determined in Step 7.4.2. The concentration of a specific analyte is calculated as follows:

Aqueous samples

$$\text{Concentration (ug/L)} = [(A_x)(A)(V_t)(D)]/[(A_s)(V_i)(V_s)]$$

where:

A_x = Response for the analyte in the sample, units may be in area counts or peak height.

A = Amount of standard injected or purged, ng.

A_s = Response for the external standard, units same as for A_x.

V_i = Volume of extract injected, uL. For purge-and-trap analysis, V_i is not applicable and therefore = 1.

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.

V_t = Volume of total extract, uL. For purge-and-trap analysis, V_t is not applicable and therefore = 1.

V_s = Volume of sample extracted or purged, mL.

Nonaqueous samples

$$\text{Concentration (ng/g)} = [(A_x)(A)(V_t)(D)]/[(A_s)(V_i)(W)]$$

generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.2 Before processing any samples, the analyst should demonstrate, through the analysis of a reagent blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent water blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement steps.

8.3 For each analytical batch (up to 20 samples), a reagent blank, matrix spike, and replicate or matrix spike replicate must be analyzed (the frequency of the spikes may be different for different monitoring programs). The blank and spiked samples must be carried through all stages of the sample preparation and measurement steps.

8.4 The experience of the analyst performing gas chromatography is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration sample should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal?; Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), recalibration of the system must take place.

8.5 Required instrument QC

8.5.1 Step 7.4 requires that the %RSD vary by < 20% when comparing calibration factors to determine if a five point calibration curve is linear.

8.5.2 Step 7.4 sets a limit of ± 15% difference when comparing daily response of a given analyte versus the initial response. If the limit is exceeded, a new standard curve must be prepared.

8.5.3 Step 7.5 requires the establishment of retention time windows.

8.5.4 Step 7.6.8 sets a limit of ± 15% difference when comparing the initial response of a given analyte versus any succeeding standards analyzed during an analysis sequence.

8.5.5 Step 7.6.9.2 requires that all succeeding standards in an analysis sequence must fall within the daily retention time window established by the first standard of the sequence.

8.6.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to Step 8.6.6.1 or 8.6.6.2.

8.6.6.1 Locate and correct the source of the problem and repeat the test for all analytes of interest beginning with Step 8.6.2.

8.6.6.2 Beginning with Step 8.6.2, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Step 8.6.2.

8.7 The laboratory must, on an ongoing basis, analyze a reagent blank and a matrix spiked replicate for each analytical batch (up to a maximum of 20 samples/batch) to assess accuracy. For soil and waste samples where detectable amounts of organics are present, replicate samples may be appropriate in place of spiked replicates. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.7.1 The concentration of the spike in the sample should be determined as follows:

8.7.1.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Step 8.7.2, whichever concentration would be larger.

8.7.1.2 If the concentration of a specific analyte in a water sample is not being checked against a limit specific to that analyte, the spike should be at the same concentration as the QC reference sample (Step 8.6.2) or 1 to 5 times higher than the background concentration determined in Step 8.7.2, whichever concentration would be larger. For other matrices, the recommended spiking concentration is 20 times the PQL.

8.7.1.3 For semivolatile organics, it may not be possible to determine the background concentration levels prior to spiking (e.g. maximum holding times will be exceeded). If this is the case, the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or the QC reference sample concentration (Step 8.6.2). For other matrices, the recommended spiking concentration is 20 times the PQL.

8.7.2 Analyze one unspiked and one spiked sample aliquot to determine percent recovery of each of the spiked compounds.

8.7.2.1 Volatile organics - Analyze one 5-mL sample aliquot to determine the background concentration (B) of each analyte. If necessary, prepare a new QC reference sample concentrate (Step 8.6.1)

8.8.1 Preparation of the QC check sample - For volatile organics, add 10 uL of the QC check sample concentrate (Step 8.6.1 or 8.7.2) to 5 mL of water. For semivolatile organics, add 1.0 mL of the QC check sample concentrate (Step 8.6.1 or 8.7.2) to 1 L of water. The QC check sample needs only to contain the analytes that failed criteria in the test in Step 8.7. Prepare the QC check sample for analysis following the guidelines given in Method 3500 (e.g. purge-and-trap, extraction, etc.).

8.8.2 Analyzed the QC check sample to determine the concentration measured (A) of each analyte. Calculate each percent recovery (p_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.8.3 Compare the percent recovery (p_s) for each analyte with the corresponding QC acceptance criteria found in the appropriate Table in each of the methods. Only analytes that failed the test in Step 8.7 need to be compared with these criteria. If the recovery of any such analyte falls outside the designated range, the laboratory performance for that analyte is judged to be out of control, and the problem must be immediately identified and corrected. The result for that analyte in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.9 As part of the QC program for the laboratory, method accuracy for each matrix studied must be assessed and records must be maintained. After the analysis of five spiked samples (of the same matrix type) as in Step 8.7, calculate the average percent recovery ($\bar{p}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{p} - 2s_p$ to $\bar{p} + 2s_p$. If $\bar{p}$ = 90% and s_p = 10%, for example, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each analyte on a regular basis (e.g. after each five to ten new accuracy measurements).

8.10 To determine acceptable accuracy and precision limits for surrogate standards the following procedure should be performed.

8.10.1 For each sample analyzed, calculate the percent recovery of each surrogate in the sample.

8.10.2 Once a minimum of thirty samples of the same matrix have been analyzed, calculate the average percent recovery (p) and standard deviation of the percent recovery (s) for each of the surrogates.

8.10.3 For a given matrix, calculate the upper and lower control limit for method performance for each surrogate standard. This should be done as follows:

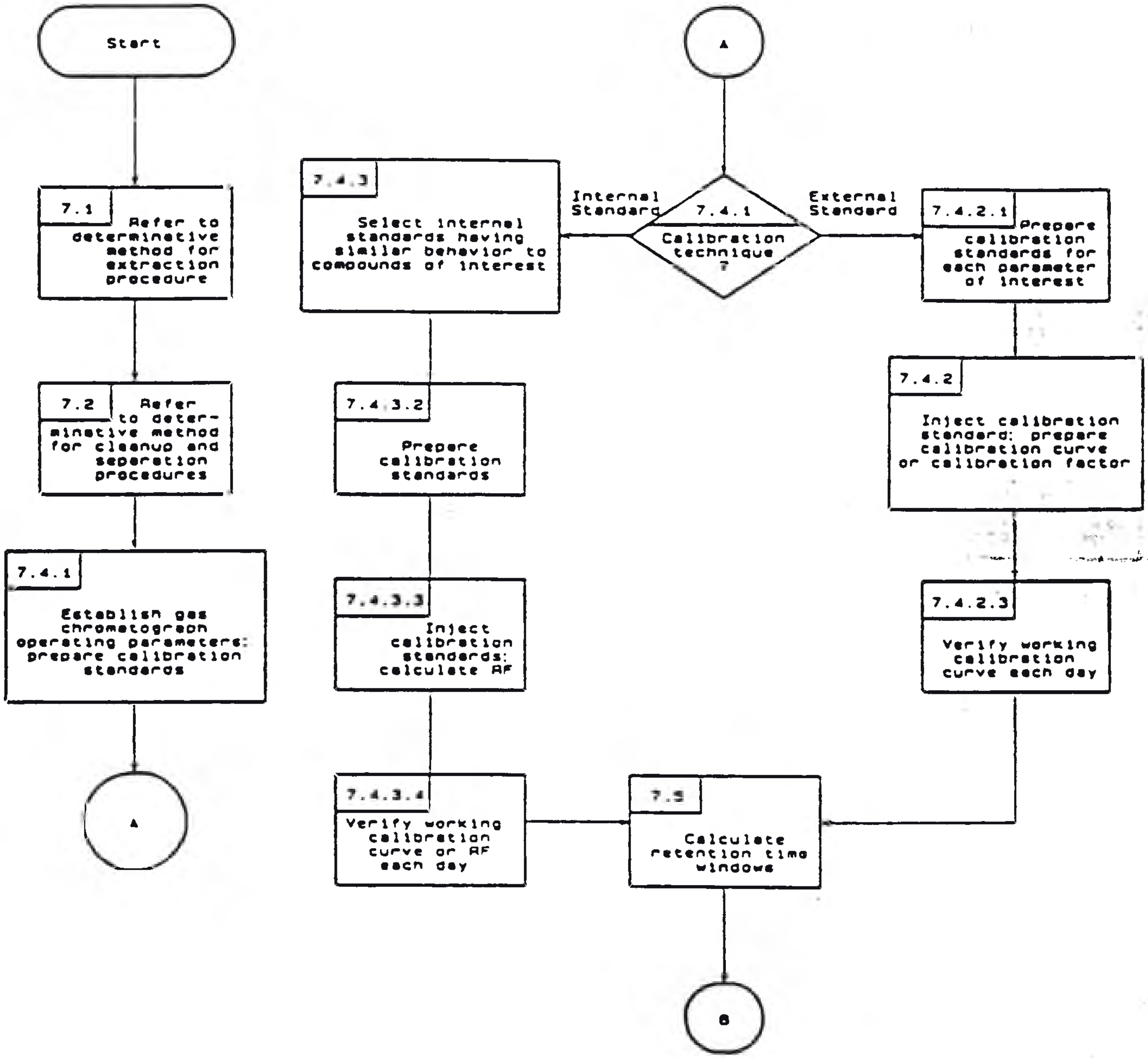
Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

8.10.4 For aqueous and soil matrices, these laboratory established surrogate control limits should, if applicable, be compared with the control limits listed in Tables A and B of Methods 8240 and 8270,

3. U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, July 1985, Revision.

4. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

5. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.


Start
7.1
Refer to determinative method for extraction procedure
7.2
Refer to determinative method for cleanup and separation procedures
7.4.1
Establish gas chromatograph operating parameters: prepare calibration standards
A
A
7.4.1
Calibration technique ?
Internal Standard
External Standard
7.4.3
Select internal standards having similar behavior to compounds of interest
7.4.3.2
Prepare calibration standards
7.4.3.3
Inject calibration standards: calculate RF
7.4.3.4
Verify working calibration curve or RF each day
7.4.2.1
Prepare calibration standards for each parameter of interest
7.4.2
Inject calibration standard: prepare calibration curve or calibration factor
7.4.2.3
Verify working calibration curve each day
7.5
Calculate retention time windows
B

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region 10

B. Authorized By: Bruce Woods (206) 553-1193

C. Prepared By: Laura CastrillI (206) 553-4323

D. Date of Request: March 8, 1991

E. Site Name: Ridgefield Brick and Tile (RBT)
Ridgefield, Washington

F. 2 digit Superfund site identifier: N/A

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

The requested analytical service will be used to determine the concentration of chromium and arsenic in ground water (System No. 1) and leachate (System No. 2) at RBT.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):

This will be a split-sampling event.

Six unfiltered aqueous environmental samples, two unfiltered aqueous duplicate samples, and two unfiltered aqueous equipment rinsate field blanks will be received to analyze for total arsenic and chromium (**a total of 10 unfiltered samples/blanks**). From previous sampling data, these unfiltered samples and blanks are expected to contain low concentrations of arsenic and chromium.

Similarly, six filtered aqueous environmental samples, two filtered aqueous duplicate samples, and two filtered aqueous equipment rinsate field blanks will be received to analyze for dissolved arsenic and chromium (**a total of 10 filtered samples/blanks**). From previous sampling data, these filtered samples and blanks are expected to contain low concentrations of arsenic and chromium.

3. **Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):**

   The purpose of this analysis will be to supply supporting analytical data to EPA to aid in characterizing the RBT site. EPA is considering a clean closure option for the RBT landfill.

4. **Estimated date(s) of collection:** March 27 and 28, 1991

5. **Estimated date(s) and method of shipment:**

   Samples will be shipped by Federal Express on March 27 and 28, 1991 following each day's sampling activities.

6. **Number of days analysis and data required after laboratory receipt of samples:**

   Samples received to analyze for dissolved chromium and arsenic should be field-filtered and preserved with HNO_3 to a pH of <2. Those samples taken to analyze for total chromium and arsenic should also be preserved with HNO_3 to a pH of <2. Analysis should occur within 6 months of collection.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

   The protocols used for these analyses have been taken from Methods for Chemical Analysis of Water and Wastes (EPA-600/4-79-020). These include Method 218.2 for chromium and Method 206.2 for arsenic. Copies of these methods are attached to this SAS request.

8. **Special technical instructions (if outside protocol requirements, specify compound names, SAS numbers, detection limits, etc.):**

   No special techniques will be required other than those specified by Methods 218.2 and 206.2.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.**

   Data will be reported in the standard Contract Laboratory program Routine Analytical Service format.

11. **Name of sampling/shipping contact:**

Gary Bruno
PRC Environmental Management, Inc.

Phone: 206/624-2692

12. **Data Requirements**

Parameter	Quantitation Limit	Precision (percent or Concentration)
Arsenic	1 µg/L	± 10
Chromium	1 µg/L	± 10

13. **QC Requirements**

Audits required	Frequency of Audits	Limits (percent or Concentration)
Method Blanks	Once per 20 samples or once per 12 hours of continuous assay.	± 10 %
Analytical System Preparation	Instrument parameters are outlined in each method.	See Methods 218.2 and 206.2.
Matrix Duplicate	Once per 20 samples	± 10 %
Laboratory Duplicates	Once per 20 samples	± 10 %

14. **Action Required if Limits are Exceeded**

Recalibrate analysis instrument as indicated and reassay or call Bruce Woods, QA chemist, at 206-553-1193 (FTS 399-1193) or Gerald Muth, CLP DPO, at 206-871-0748 (FTS 390-1282), immediately, for problem resolution.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

ARSENIC

Method 206.2 (Atomic Absorption, furnace technique)

STORET NO. Total 01002
Dissolved 01000
Suspended 01001

Optimum Concentration Range: 5–100 ug/1
Detection Limit: 1 ug/1

Preparation of Standard Solution

1. Stock solution: Dissolve 1.320 g of arsenic trioxide, As_2O_3 (analytical reagent grade) in 100 ml of deionized distilled water containing 4 g NaOH. Acidify the solution with 20 ml conc. HNO_3 and dilute to 1 liter. 1 ml = 1 mg As (1000 mg/1).

2. Nickel Nitrate Solution, 5%: Dissolve 24.780 g of ACS reagent grade $Ni(NO_3)_2 \cdot 6H_2O$ in deionized distilled water and make up to 100ml.

3. Nickel Nitrate Solution, 1%: Dilute 20 ml of the 5% nickel nitrate to 100 ml with deionized distilled water.

4. Working Arsenic Solution: Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Withdraw appropriate aliquots of the stock solution, add 1 ml of conc. HNO_3, 2ml of 30% H_2O_2 and 2ml of the 5% nickel nitrate solution. Dilute to 100 ml with deionized distilled water.

Sample Preservation

1. For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.

Sample Preparation

1. Transfer 100 ml of well-mixed sample to a 250 ml Griffin beaker, add 2 ml of 30% H_2O_2 and sufficient conc. HNO_3 to result in an acid concentration of 1%(v/v). Heat for 1 hour at 95°C or until the volume is slightly less than 50 ml.

2. Cool and bring back to 50 ml with deionized distilled water.

3. Pipet 5 ml of this digested solution into a 10-ml volumetric flask, add 1 ml of the 1% nickel nitrate solution and dilute to 10 ml with deionized distilled water. The sample is now ready for injection into the furnace.

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NOTE: If solubilization or digestion is not required, adjust the HNO_3 concentration of the sample to 1% (v/v) and add 2 ml of 30% H_2O_2 and 2 ml of 5% nickel nitrate to each 100 ml of sample. The volume of the calibration standard should be adjusted with deionized distilled water to match the volume change of the sample.

Instrument Parameters (General)

1. Drying Time and Temp: 30 sec–125°C.
2. Ashing Time and Temp: 30 sec–1100°C.
3. Atomizing Time and Temp: 10 sec–2700°C.
4. Purge Gas Atmosphere: Argon
5. Wavelength: 193.7 nm
6. Other operating parameters should be set as specified by the particular instrument manufacturer.

Analysis Procedure

1. For the analysis procedure and the calculation, see "Furnace Procedure" part 9.3 of the Atomic Absorption Methods section of this manual.

Notes

1. The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 ul injection, purge gas interrupt and non-pyrolytic graphite. Smaller size furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.
2. The use of background correction is recommended.
3. For every sample matrix analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
4. If method of standard addition is required, follow the procedure given earlier in part 8.5 of the Atomic Absorption Methods section of this manual.
5. For quality control requirements and optional recommendations for use in drinking water analyses, see part 10 of the Atomic Absorption Methods section of this manual.
6. Data to be entered into STORET must be reported as ug/1.

Precision and Accuracy

1. In a single laboratory (EMSL), using a mixed industrial-domestic waste effluent containing 15 ug/1 and spiked with concentrations of 2, 10 and 25 ug/1, recoveries of 85%, 90% and 88% were obtained respectively. The relative standard deviation at these concentrations levels were ±8.8%, ±8.2%, ±5.4% and ±8.7%, respectively.
2. In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at concentrations of 20, 50 and 100 ug As/1, the standard deviations were ±0.7, ±1.1 and ±1.6 respectively. Recoveries at these levels were 105%, 106% and 101%, respectively.

CHROMIUM

Method 218.2 (Atomic Absorption, furnace technique)

STORET NO. 01034
Dissolved 01030
Suspended 01031

Optimum Concentration Range: 5–100 ug/l
Detection Limit: 1 ug/l

Preparation of Standard Solution

1. Stock solution: Prepare as described under "direct aspiration method".
2. Calcium Nitrate Solution: Dissolve 11.8 grams of calcium nitrate, $Ca(NO_3)_2 \cdot 4H_2O$ (analytical reagent grade) in deionized distilled water and dilute to 100 ml. 1 ml = 20 mg Ca.
3. Prepare dilutions of the stock chromium solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared to contain 0.5% (v/v) HNO_3. To each 100 ml of standard and sample alike, add 1 ml of 30% H_2O_2 and 1 ml of the calcium nitrate solution.

Sample Preservation

1. For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.

Sample Preparation

1. Prepare as described under "direct aspiration method". Sample solutions for analysis should contain 0.5% v/v HNO_3.

Instrument Parameters (General)

1. Drying Time and Temp: 30 sec–125°C.
2. Ashing Time and Temp: 30 sec–1000°C.
3. Atomizing Time and Temp: 10 sec–2700°C.
4. Purge Gas Atmosphere: Argon
5. Wavelength: 357.9 nm
6. Other operating parameters should be set as specified by the particular instrument manufacturer.

Analysis Procedure

1. For the analysis procedure and the calculation, see "Furnace Procedure" part 9.3 of the Atomic Absorption Methods section of this manual.

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Notes

1. The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 ul injecton, continuous flow purge gas and non-pyrolytic graphite.
2. Hydrogen peroxide is added to the acidified solution to convert all chromium to the trivalent state. Calcium is added to a level above 200 mg/1 where its suppressive effect becomes constant up to 1000 mg/1.
3. Background correction may be required if the sample contains high dissolved solids.
4. Nitrogen should not be used as a purge gas because of possible CN band interference.
5. Pipet tips have been reported to be a possible source of contamination. (See part 5.2.9 of the Atomic Absorption Methods section of this manual.)
6. For every sample matrix analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
7. If method of standard addition is required, follow the procedure given earlier in part 8.5 of the Atomic Absorption Methods section of this manual.
8. For quality control requirements and optional recommendations for use in drinking water analyses, see part 10 of the Atomic Absorption Methods section of this manual.
9. Data to be entered into STORET must be reported as *u*g/1.

Precision and Accuracy

1. In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at concentrations of 19, 48, and 77 *u*g Cr/1, the standard deviations were ±0.1, ±0.2, and ±0.8, respectively. Recoveries at these levels were 97%, 101%, and 102%, respectively.

APPENDIX B

OPERATORS MANUAL FOR THE

HNu PHOTOIONIZATION DETECTOR

MODEL P-101

OPERATIONAL PROCEDURE FOR

HNu MODEL PI 101

PHOTOIONIZATION ANALYZER

PREPARED BY

CHENG-WEN TSAI, CHEMIST

QUALITY ASSURANCE OFFICE
U.S.EPA, REGION V

TABLE OF CONTENT

OPERATION PROCEDURE FOR
HNu MODEL PI 101
PHOTOIONIZATION ANALYZER

I. INTRODUCTION

1.0 Operation Principle

The HNu Model 101 photoionization detector has been designed to measure the concentration of trace gases in many industrial or plant atmospheres. The instrument has similar capabilities outdoors. The analyzer employs the principle of photoionization for detection. This process is termed photoionization because the absorption of ultraviolet light (a photon) by a molecule leads to ionization via:

$$RH + hv \text{ ---------------> } RH^+ + e^-$$

where RH = trace gas
hv = a photon with an energy greater than or equal to an ionization potential of RH.

The sensor consists of a sealed ultraviolet light source that emits photons which are energetic enough to ionize many trace species (particularly organics), but do not ionize the major components of air such as O_2, N_2, CO, CO_2 or H_2O. A chamber adjacent to the ultraviolet light source contains a pair of electrodes. When a positive potential is applied to one electrode, the field created drives any ions, formed by abosrption of UV light, to the collector electrode where the current (proportional to concentration) is measured. The useful range of the instrument is from a fraction of a ppm to about 2,000 ppm.

2.0 Instrument Sensitivity and Calibration

The instrument responds to atmospheric compounds with ionization potentials equal to or less than the ionization energy of the UV light source. If a compound in air has an ionization potential greater than the energy source of the lamp, it will not be detected. Table 1 presents organic and inorganic compounds and the light sources that should be used to detect each compound. The instrument is capable of using 1 of the 3 light sources - 9.5, 10.2, and 11.7 ev lamps. In addition, not all compounds respond equally to each light sources and thus they vary in their sensitivity to ionization. As a result of varying sensitivities to photoionization, the response given by the instrument may or may not reflect the actual atmospheric concentration of the compound being detected. Table 2 represents the relative sensitivities for various gases relative to a 10.2 ev light source. Use this table to determine the approximate response of the instrument to a compound of interest, and to select the appropriate light (lamp) source.

TABLE 1 LAMP SOURCE IONIZATION POTENTIALS FOR ORGANIC AND INORGANIC AIRBORNE COMPOUNDS

9.5 eV Lamp Source

Acids (organic)
Alcohols
Amines
Aniline
Aromatics
Benzene
Borontribromide
Chlorinated aromatics
Dimethyldisulfide
Dimethylsulfide
Ketones
Phenol
Pyridine
Styrene
Toluene

10.2 eV Lamp Source

Acetaldehyde
Acetic acid
Acetone
Acids (organic)
Acrolein (acelyates)
Alcohols
Aldehydes
Aliphatics
Alkyl halides
Allyl alcohol
Amides
Amines
Ammonia
Aniline
Aromatics
Arsine
Asphalt emissions
Benzene
Bromine
Butane
Boron tribromide
Carbon disulfide
Chlorinated aromatics
Chlorinated hydrocarbons
Chloropenes
Cyclohexanane
Dibromochoropropene
Dichloroporpylene
Dimethyl disulfide
Dimethyl formal dehyde
Dimethyl sulfide
Epichlorhydrin
Esters
Ethanol
Ethyl methacrylate
Ethylene
Ethylene dibromide
Ethylene imine
Ethylene oxide
Furan
Heterocyclics
Hexane
Hexamethyl phosphoric triamide
Hydrazine
Hydrogen sulfide
Hydrogen selenide

TABLE 1 (CONTINUED)

10.2 eV Lamp Source (Cont'd.)	
Iodine vapor	Phosphine
Isopropanol	Phosphorus trichloride
Ketones	Picolines
Lutidines	Pinene
Methyl bromide	Propylene
Methyl isocyanate	Pyridine
Methyl mercaptan	Pyrole
Methyl methacrylate	Styrene
Mineral spirits	Tetrahydrofuran
Naptha	Tetraethyl lead
Nitrates	Thionyl chloride
Nitrites	Toluene
Nitro alkanes	Vinyl acetate
Nitro benzene	Vinyl bromide
N-Octane	Vinyl chloride
Olefins	Vinylidine chloride
Phenol	
Phostoxin	

11.7 Lamp Source	
Acetic anhydride	Formic acid
Acetylene	Methanol
Acrylonitrile	Methylene chloride
Alcohols	Nitrates
Aldehydes	Nitrites
Alphatics	Nitro alkanes
Alkyl halides	Phostoxin
Butane	Propane
Carbon tetrachloride	Serafume
Chloroform	
Ethane	
Ethylene dichloride	
Formaldehyde	

TABLE 2 RELATIVE SENSITIVITIES FOR VARIOUS GASES
(10.2 eV Lamp)

Species	Photoionization Sensitivity*
p-xylene	11.4
m-xylene	11.2
benzene	10.0 (reference standard)
toluene	10.0
diethyl sulfide	10.0
diethyl amine	9.9
styrene	9.7
trichloroethylene	8.9
carbon disulfide	7.1
isobutylene	7.0
acetone	6.3
tetrahydrofuran	6.0
methyl ethyl ketone	5.7
methyl isobutyl ketone	5.7
cyclohexanone	5.1
naptha (85% aromatics)	5.0
vinyl chloride	5.0
methyl isocyanate	4.5
iodine	4.5
methyl mercaptan	4.3
dimethyl sulfide	4.3
allyl alcohol	4.2
propylene	4.0
mineral spirits	4.0
2,3-dichloropropene	4.0
cyclohexene	3.4
crotonaldehyde	3.1
acrolein	3.1
pyridine	3.0
hydrogen sulfide	2.8
ethylene dibromide	2.7
n-octane	2.5
acetaldehyde oxime	2.3
hexane	2.2
phosphine	2.0
heptane	1.7
allyl chloride (3-chloropropene)	1.5
ethylene	1.0
ethylene oxide	1.0
acetic anhydride	1.0
a-pinene	0.7
dibromochloropropane	0.7
epichlorohydrin	0.7
nitric oxide	0.6

TABLE 2 RELATIVE SENSITIVITIES FOR VARIOUS GASES (10.2 eV Lamp) (Continued)

Species	Photoionization Sensitivity*
b-pinene	0.5
citral	0.5
ammonia	0.3
acetic acid	0.1
nitrogen dioxide	0.02
methane	0.0
acetylene	0.0

*Expressed in ppm (v/v).

There are two types of operations that are used for calibration. For Type 1 Operation, a non-regulatory (or non-target) compound such as isobutylene is used for calibration. In this case, the instrument reading is reported in terms relative to the calibration compound used for calibration. For the type 2 operation, the target compound or compounds are used for calibration. As a result, the instrument is calibrated to respond directly in ppm by volume of the target compound(s).

3.0 Instrument Specifications

3.1 Performance

- O Range : 0.1 to 2000 ppm
- O Detection Limit : 0.1 ppm
- O Sensitivity (max.) : 0 to 2 ppm FSD over 100 division meter scale
- O Repeatability : ± 1% of FSD
- O Linear Range : 0.1 to 600 ppm
- O Useful Range : 0.1 to 2000 ppm
- O Response Time : less than 3 seconds to reach 90% full scale
- O Ambient humidity : up to 95% relative humidity
- O Operating Temperature : Ambient to 40°C (instrument is temperature compensated so that a 20°C change in temperature corresponds to a change in reading of ± 2% full scale at maximum sensitivity.

3.2 Power Requirements and Operating Times

- O Continuous use on battery : approximately 10 hours
- O Continuous use with HNu recorder reduces instrument battery operating time to approximately 5 hours
- O Recharge time : less than 14 hours; a 3 hours charge will charge up to 90% full charge
- O Recharge Current : maximum 0.4 amps at 15 VDC

II. OPERATIONAL PROCEDURE

1.0 Instrument Check-Out

1.1 Remove instrument box cover by pulling up on fasteners.

1.2 On the instrument panel, there will be a label containing information on light source, calibration date, calibration gas, and span setting.

1.2.1 If the instrument has not been calibrated in the last 14 days or since its last field use, it should be re-calibrated. Check the instrument log, which should be maintained with the instrument, for the instrument status and its calibration history. For general use, the instrument should be calibrated to isobutylene at a span setting of 9.8.

1.2.2 Check the label for light source and refer to Table 1 for ionization potentials of various compounds. If the compound you wish to detect is not listed for the light sources provided with instrument, then the light source will have to be changed. Use the probe with the proper light source for the compounds to be detected.

1.2.3 Once it has been determined that the instrument has the correct lamp, the instrument may need to be recalibrated for the specific compound of interest. Use Procedure under 2.1.3 of this Section to calibrate the instrument.

1.2.4 Check the battery supply by connecting the probe to the instrument box, and turning the function switch to the battery check position (Figure 1). (Note: The battery check indicator will not function unless the probe is attached.) The meter needle should deflect to the far right or above the green zone. If the needle is below or just within the green zone or the red LED indicator is on, the battery should be recharged. Follow the procedure described in Section III (Maintenance and Trouble shooting) to recharge the battery.

1.2.5 Repack the instrument for shipment to the field.

2.0 Field Operation

2.1 Calibration

2.1.1 **Equipment and Materials**

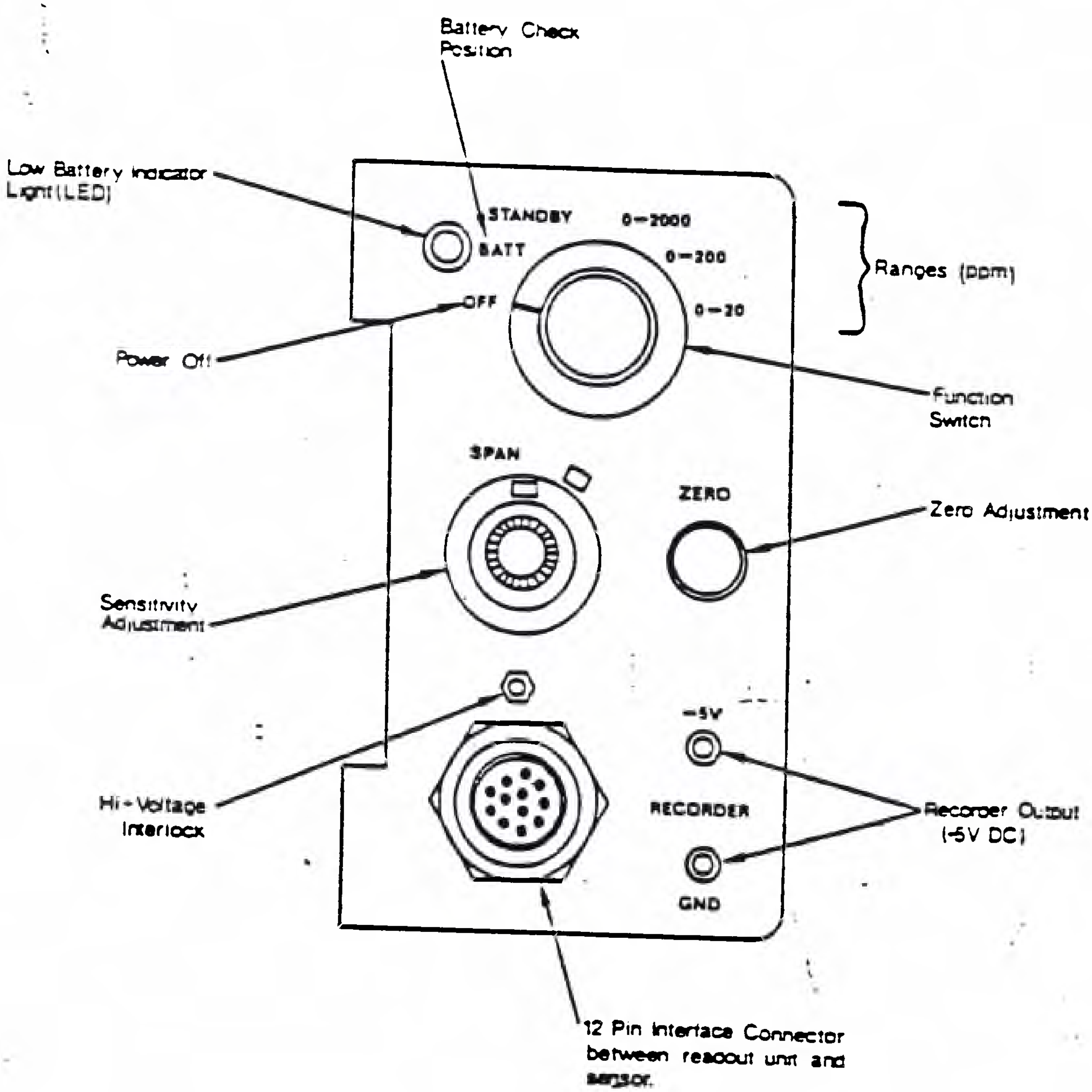


FIGURE 1 INSTRUMENT CONTROL PANEL FEATURES

- O Calibration Gas (2 ranges)

  Low range 0-20 ppm and mid-range 20-200 ppm of isobutylene gas are used for standard field operation when contaminants are unknown or a mixture of gases is present. The isobutylene gas is used for general calibration because of the instrument's relatively high sensitivity to it and the non-toxic nature of the gas.
  Note: A specialty gas may be required if a single atmospheric contaminant is present and the contaminant has a sensitivity different from that of the calibration gas (isobutylene).

- O Tubing and fittings (see Figure 2).

- O Rotometer or bubble flow meter.

- O Field Log, calibration form, and data reporting form.

- O Table 1 for ionization potentials for compounds of interest.

2.1.2 Calibration Frequency

This instrument should be calibrated after each field use and prior to each field use. Continuous calibration check should be performed frequently during field operation (for example, check the instrument zero and calibration after every 10 measurements) and document the results properly. Caution: Do Not Change the Settings.

2.1.3 Calibration Procedure

2.1.3.1 Use a three-points procedure to facilitate the proper instrument calibration over appropriate operating ranges. Distinct mixtures of calibration gas with known concentration for selective operating range should be used for calibration. Each mixture should give a 3/4 scale deflection in its respective operating range.

2.1.3.2 Instrument Setup.

Step 1: Remove Instrument cover by pulling up on the side straps.

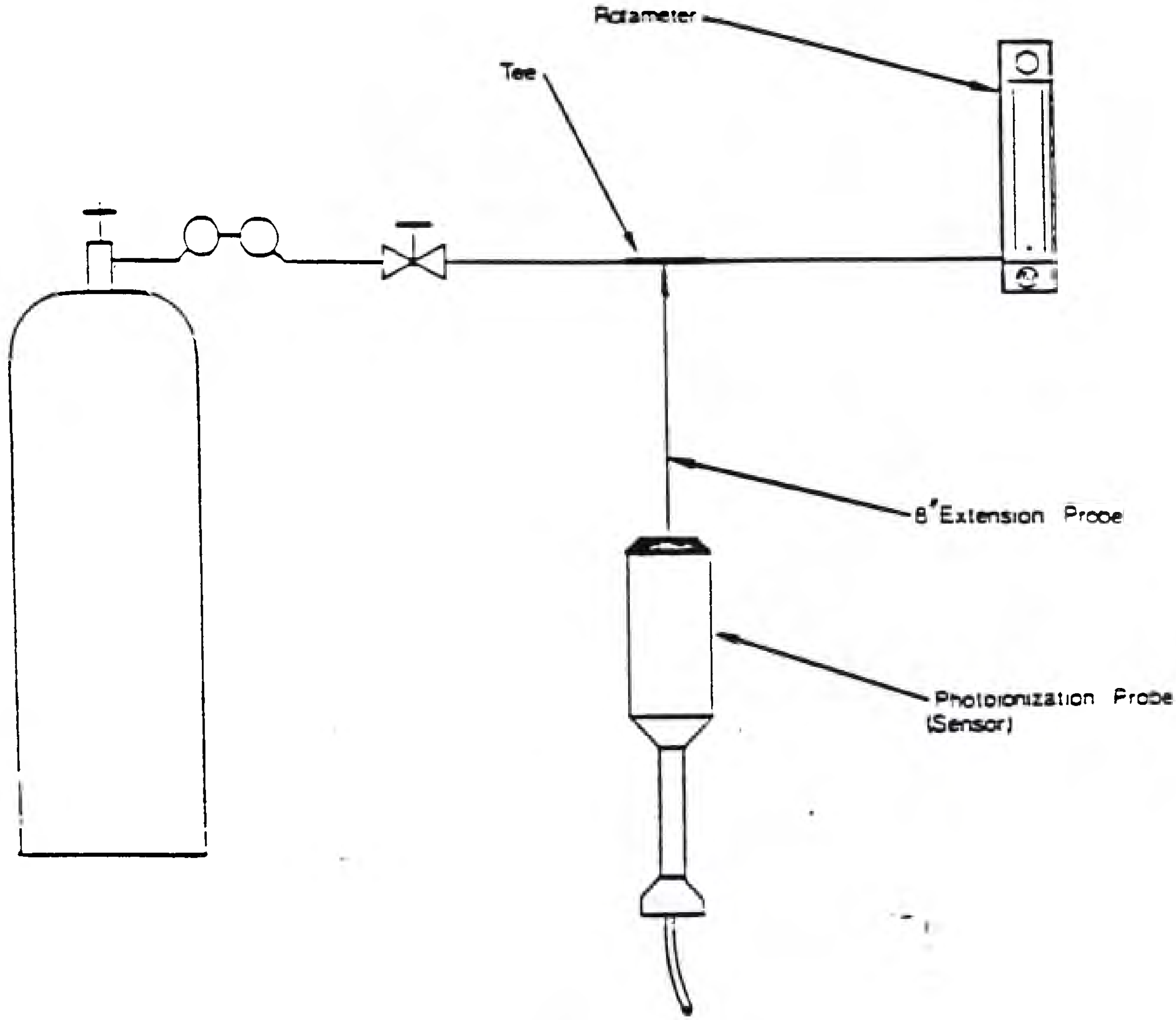


FIGURE 2 RECOMMENDED CALIBRATION PROCEDURE FOR PHOTOIONIZATION ANALYZER

Step 2: Prior to calibration, check the function switch (Figure 1) on the control panel to make sure it is in the OFF position. The probe nozzle is stored inside the instrument cover. Remove cover plate by pulling up on the pins that fasten the cover plate.

Step 3: Remove the nozzle from the cover. Assemble probe by screwing nozzle into casing.

Step 4: Attach probe cable to instrument box inserting 12 pin interface connector of the probe cable into the connector on the instrument panel. Match the alignment keys and insert connector. Turn connector in clockwise direction until a distinct snap and lock is felt.

Step 5: Turn the function switch to the Battery Check position. When the battery is charged, the needle should read within or above the green battery arc on the scale plate. If the needle is below the green arc or the red LED light comes on, the instrument should be recharged prior to making any measurements. Implement steps in Section III to recharge battery.

Step 6: Turn the function switch to the ON position. In this position, the UV light source should be on. To verify, gaze at the end of the probe for a purple glow. Do Not Look Directly at the Lamp Itself. If the lamp does not come on refer to Maintenance Step in 2.2 (Section III).

Step 7: To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counter clockwise rotation yields a downscale deflection. (Note: No zero gas is needed since this is an electronic zero adjustment.) If the span adjustment is changed during instrument calibration, the zero should be rechecked and adjusted. If necessary, wait 15 to 20 seconds to ensure that the zero reading is stable. Readjust as necessary.

2.1.3.3 Calibration Steps

Step 1: Insert one end of T tube (Figure 2) into probe. Insert second end of probe into calibration gas in the 20-200 ppm range. The third end of probe should have the rotometer (bubble meter) attached.

Step 2: Set the function switch in the 0-200 ppm range. Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotometer. The instrument will draw in the volume required for detection with the rotometer indicating excess flow.

Step 3: Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas. (Calibration gas value is labeled on the cylinder).

Step 4: Turn instrument switch to the standby position and check the electronic zero. Reset zero potentiometer as necessary following step 7 of 2.1.3.2.

Step 5: Record on form and field log all original and readjusted settings as specified in the form.

Step 6: Next, set the function switch to the 0-20 ppm. Remove the mid-range (20-200 ppm) calibration gas cylinder and attach the low range--(0-20 ppm) calibration gas cylinder as described above.

Step 7: Do not adjust the span potentiometer. The observed reading should be +3 ppm of the concentration specified for the low range calibration gas. If this is not the case, recalibrate the mid range scale repeating Step 1 thru 6 above. If the low range reading consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. Clean window following Step 2 under 2.3 (Section III). When the observed reading is within the required tolerances, the instrument is fully calibrated.

2.2 Sample Measurement

Step 1: Place function switch in 0-20 ppm range for field monitoring. This will allow for the most sensitive, quick response in detecting airborne contaminants.

Step 2: Before entering a contaminanted area, determine background concentration. This concentration should be used as a reference to readings made in the contaminated area. Under no circumstance should one attempt to adjust the zero or span adjustments while the instrument is being operated in the field.

Step 3: Take measurements in contaminated area, recording readings and locations. Should readings exceed the 0-20 scale, switch the function switch to the 0-200 or 0-2,000 range as appropriate to receive a direct reading. Return the instrument switch to the 0-20 range when readings are reduced to that level. Record measuremeasurements in notebook or on an appropriate form.

Step 4: Keep in mind health and safety action guidelines for the level of protection you are wearing. Sustained readings above a certain level may force you to vacate an area or upgrade your level of protection.

Note: The instrument will not function properly in high humidity or when the window to the light housing is dirty. If the instrument response is erratic or lower than expected.

Step 5: When finished, use the reverse Steps 1 thru 5 of Section 2.1.3.2 (Instrument Setup) to shut down the instrument.

III MAINTENANCE AND TROUBLE-SHOOTING

1.0 Battery Recharging

1.1 The instrument should be recharged 1 hour for each hour of use or overnight for a full day's use. (The battery will last 10 hours on a full charge.)

1.2 To recharge the battery (or instrument):

1.2.1 Turn the function switch to the off position.

1.2.2 Remove the charger from the instrument top compartment.

1.2.3 Place the charger plug into the jack on the left side of the instrument box.

1.2.4 Connect the charger unit to a 120 V AC supply.

1.2.5 Check charger function by turning the instrument switch to the battery check position. The meter should go upscale if the charger is working and is correctly inserted into the jack.

1.2.6 Place instrument in instrument mode and charge for the appropriate time period.

1.2.7 Turn the instrument off following the recharge cycle. When disconnecting charger, remove from 120 V AC supply before removing the mini phone plug.

2.0 <u>General Fault Determination and Correction</u>

2.1 Battery level is low. Recharge if necessary implementing steps described under 1.0 (Section III). If the battery will not recharge, it will have to be replaced.

2.2 UV Lamp function - Gaze at sample inlet when mode switch is on an instrument function position and observe for purple glow of lamp. If the lamp does not glow in any of the three instrument function positions, it may be burned out and will have to be replaced. To replace the lamp:

1. Turn the function switch to the off position and disconnect the probe connector from the readout unit.

2. Remove the exhaust screw found near the base of the probe (Figure 3).

3. Grasp the end cap in one hand and the probe shell in the other and gently pull to separate the end cap and lamp housing from the shell.

4. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing. Care must be taken so that the ion chamber does not fall out of the end cap and the lamp does not slide out of the lamp housing.

5. Turn the end cap over in your hand and tap on the top of it; the ion chamber should fall out of it.

6. Place one hand over the top of the lamp housing and tilt slightly. The light source will slide out of the housing.

7. Replace lamp with one of same energy source as the one removed by sliding it into the housing. Note: The amplifier board and instrument circuitry are calibrated for one light energy

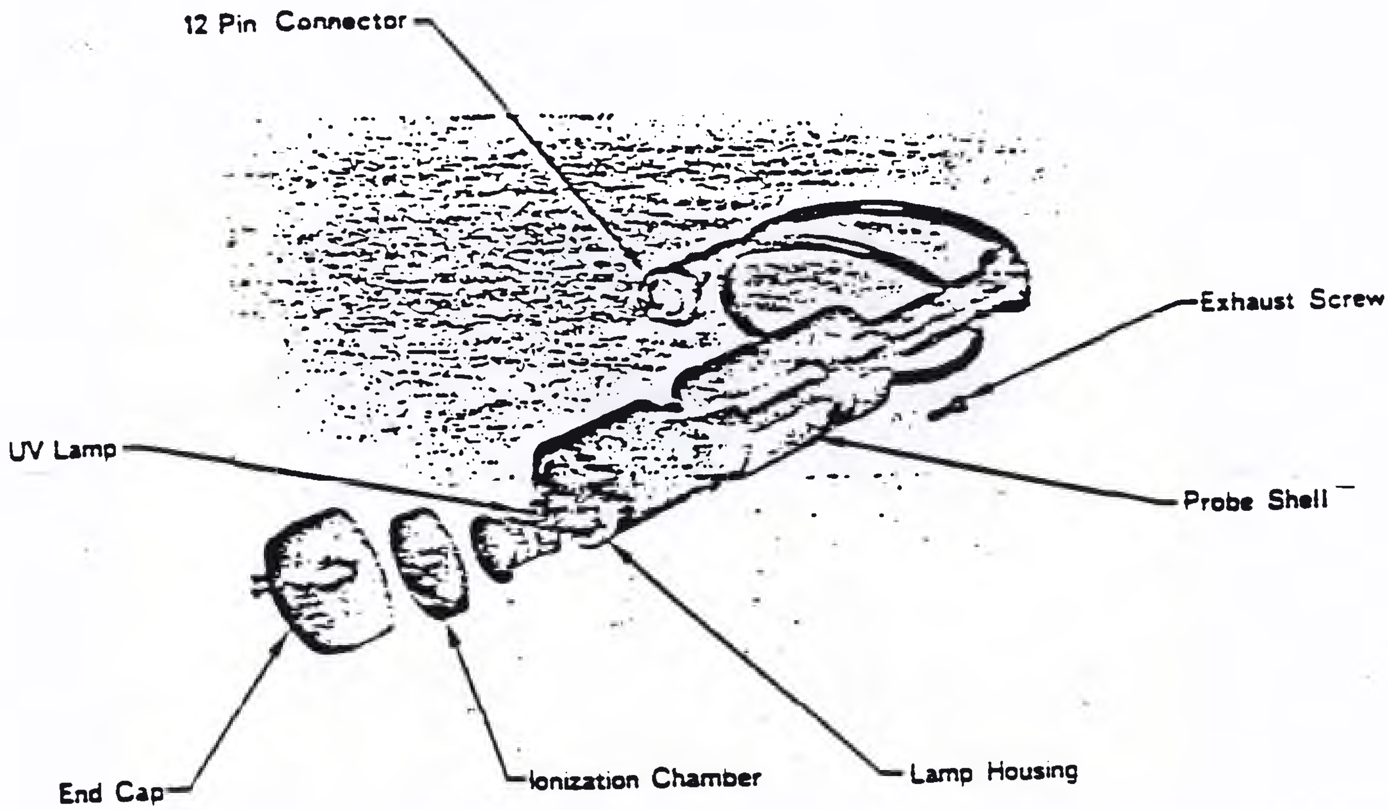


FIGURE 3 COMPONENT PARTS OF PROBE

8. Place the ion chamber on top of the lamp housing, checking to ensure that the contacts are aligned.

9. Place the end cap on top of the ion chamber and replace the two screws. The screws should be tightened only enough to seal the "O" ring. Do not overtighten.

10. Line up the pins on the base of the lamp housing with the pins inside the probe shell. Gently slide the housing assembly into the probe shell. Do not force the assembly as it only fits one way.

11. Replace and tighten the exhaust screw.

12. Reconnect the 12 pin connector and turn instrument mode switch to a function position. Check for glow of lamp. If lamp still does not function, the instrument has an electrical short or other problem that will have to be corrected at the factory.

2.3 Instrument appears to be functional, but responses are lower than expected or erratic. The window of the light source may be dirty and need to be cleaned. To clean the light source window:

1. Disassemble the probe assembly by repeating Steps 1 thru 6 under 2.2 above.

2. Clean the window of the light source using compound provided with instrument and soft clean cloth. Important: Use cleaning compound on the window of the 10.2 eV lamp only. The cleaning compound may damage the windows of the 9.5 and 11.7 eV lamps.

3. Reassemble the probe assembly repeating Step 7 through 12 above.

3.0 <u>Specific Faults</u>

3.1 No meter response in any switch position (including BATT CHK)

1. Broken meter movement: Tip instrument rapidly from side to side. Meter needle should move freely, and return to zero.

2. Electrical connection to meter is broken: Check all wires leading to meter and clean the contacts of quick-disconnects.

3. Battery is completely dead: Disconnect battery and check voltage with a volt-ohm meter.

4. Check 2 amp fuse.

5. If none of the above solves the problem, consult the factory.

3.2 Meter responds in BATT CHK position, but reads zero or near zero for all others.

1. Power supply defective: Check power supply voltages per Figure 4. If any voltage is out of specification, consult the factory.

2. Input transistor or amplifier has failed: Rotate zero control; meter should deflect up/down as control is turned. Open probe; both transistors should be fully seated in sockets.

3. Input signal connection broken in probe or readout: Check input connector on printed circuit board. Should be firmly pressed down. Check components on back side of printed circuit board. All connections should be solid, and no wires should touch any other object. Check all wires in readout for solid connections.

3.3 Instrument responds correctly in BATT CHK, and STBY, but not in measuring mode.

1. Check to see the light source is on (See Section 2.2).

2. Check high voltage power supply (See Figure 4).

3. Open end of probe, remove lamp and check high voltage on lamp contact ring.

4. If high voltage is present at all above points, light source has most likely failed. Consult the factory.

3.4 Instrument responds correctly in all positions, but signal is lower than expected.

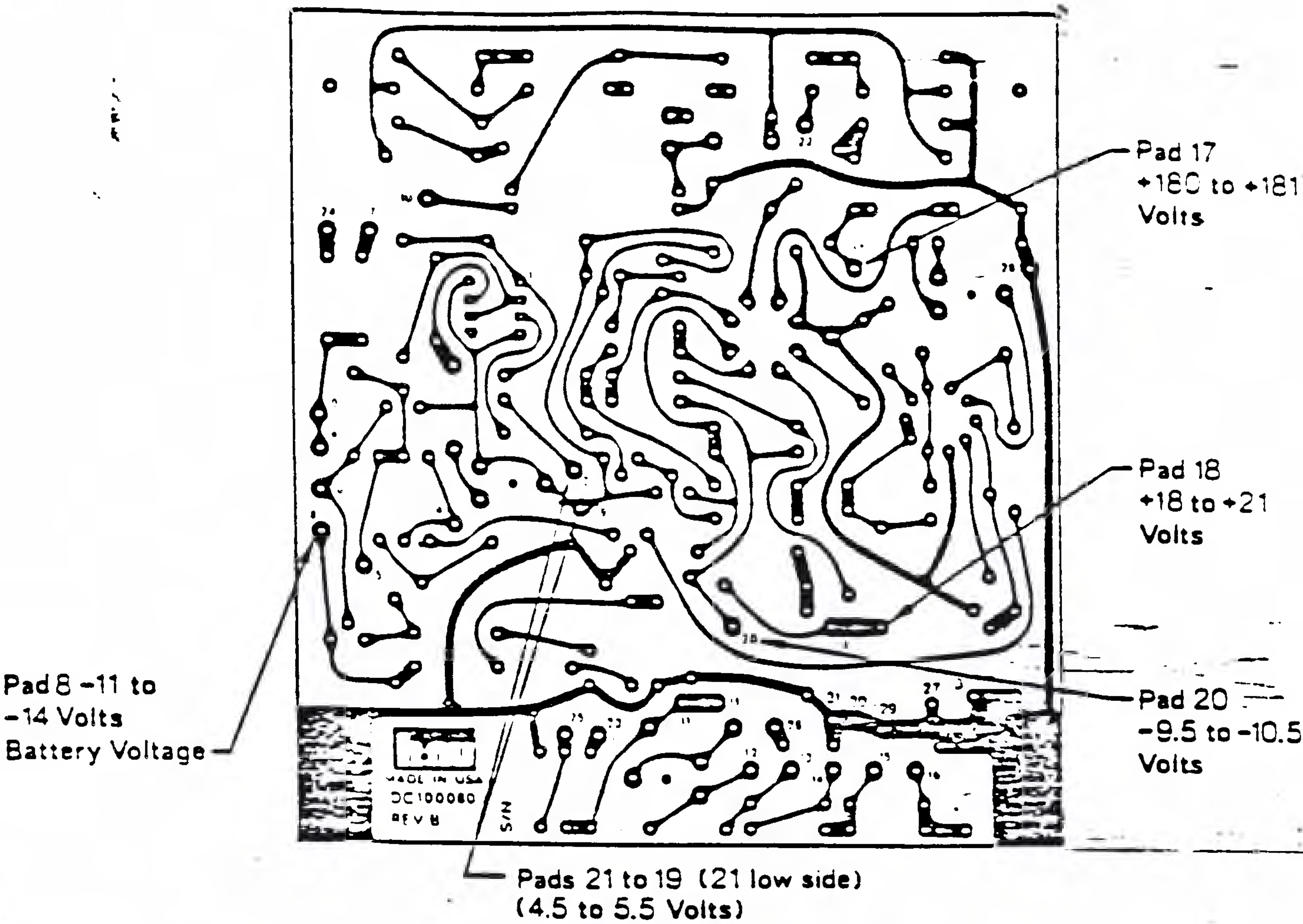
1. Check span setting for correct value.

2. Clean window of light source (See 2.3).

3. Double check preparation of standards.

4. Check power supply 180 V output. See Figure 4.

5. Check for proper fan operation. Check fan voltage. See Figure 4.



All Voltages Respect to Ground							
pads	voltage	pads	voltage	pads	voltage	pads	voltage
1	− 5.7V	9	− 12.2V	17	180V	25	0
2	GRD	10	− 12.1V	18	+ 19.4V	26	0
3	GRD	11	0	19	− 10.6V	27	GRD
4	−10 7V	12	0	20	− 9.7V	28	0
5	− 11.3V	13	0	21	− 14.5V	29	GRD
6	− 12.1V	14	0	22	− 400V	30	GRD
7	0	15	0	23	0	31	GRD
8	− 12.2V	16	0	24	0		

Figure 4 Power Supply PC Board

6. Rotate span setting. Response should change if span pot is working properly.

3.5 Instrument responds in all switch positions, but is noisy (erratic meter movement).

1. Open circuit in feedback circuit. Consult the factory.

2. Open circuit in cable shield or probe shield. Consult the factory.

3.6 Instrument response is slow and/or irreproducible.

1. Fan operating improperly. Check fan voltage. See Figure 4.

2. Check calibration and operation.

3.7 Low battery indicator.

1. Indicator comes on if battery charge is low.

2. Indicator also comes on if ionization voltage is too high.

PRC

Planning Research Corporation

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Elsa Krauss

Questions Concerning the HNU PI 101

Below are the questions and answers regarding the HNU PI 101 that were brought up during the lunch seminar on August 2nd.

I talked to Tara Velasco (Technical Specialist), Maureen Riley (Service Department Supervisor) and Bob Purdy (National Service Manager) from HNU Systems Inc. Each answer indicates the person whom I talked to.

1. Question: Moisture seems to be a problem. The needle deflects negative.

Answer: Water vapor may collect in the ionization chamber and affect instrument sensitivity. Tara Velasco recommends allowing the instrument to equilibrate for at least one hour in the environment in which it will be used. Also, a single layer of cheese cloth may be placed over the probe inlet (or over the probe extension) to absorb water vapor and filter out dust particles before they enter the detector. This practice does not solve the contamination problems, it only minimizes them. The practice is acceptable as long as the flow of sample through the probe is not restricted.

Tara recommends cleaning the lamp and ion chamber daily (depending on use)**. In addition to cleaning the lamp and ion chamber, Maureen Riley recommends cleaning the probe extention by flushing it with acetone or methanol to remove contaminats that could be drawn into the probe and detected. All parts must be completely dry before reassembly.

2. Question: Instruments are not holding "the calibration" after they come back from service at HNU Systems Inc.. This is indicated by the need to turn the span pot knob to lower numbers and it happens whithin two or three days of service.

Answer: Maureen indicated that this is a sign of a dirty lamp and ion chamber. She stated that the span setting of 9.8 is simply a reference point, and adjusting the span setting is not an indication of instrument malfuction. As the lamp gets dirty and/or ages, it loses sensitivity; therefore, it is necessary to increase the gain of the lamp by lowering the span setting.

If the lamp and ion chamber have been cleaned, and the lamp is good, there is an internal potentiometer (R48 on the power supply board) which can be adjusted to increase the gain of the lamp*. Adjustment on this internal pot should be done on rare occasions as it is delicate.

If all the above is performed and a good calibration and sensitivity cannot be obtained, a new lamp is recommended.

3. Question: Batteries don't hold the 8 hour charge as indicated in the manual. They seem to hold an average of six hours (with the instrument running constantly or in stand by).

Answer: Maureen recommended checking the battery contacts (they may need cleaning). When not taking measurements, turn the instrument off: don't leave it on stand by unless another measurement will be taken in a few minutes.

The output of the charger is -15 VDC. This output canbe adjusted to -16 VDC*, which may help lengthen the daily use of the battery. Batteries should be charged whenever the PI 101 is not in use. The probe should be attached to the readout module, and the unit function switch should be in the OFF position.

4. Question: CH2M Hill recently recommended the calibration to be performed by transfering the gas from the canister to a Tedlar bag, then calibrate the instrument with the gas from the bag.

Answer: Jim Beringer (CH2M Hill) indicated that by transfering the gas from the canister to a bag it will be at atmospheric presure and the instrument's fan will pull the gas as opposed to being introduced by presure.

Bob Purdy indicated that it is a technique which is sometimes recommended, particularly for certain special applications. However, use of the canister is also good, and HNU does not intend to replace the canister method with use of the Tedlar bag.

* These functions should be performed by an HNU trained and certified person (such as Elsa Krauss)

** The PID responds to water with a negative singal. Presence of higher than normal amounts of water in the atmosphere (90% relative humidity) may result in inaccurate sample readings. The presence of the water means more frequent cleaning of the ion chamber may be needed to remove the water and water absorbing contaminants.

I hope that this discussion has cleared some of the concerns. If you still have some questions or more problems arrive, please do not hesitate to let me know

cc: T. Velasco, HNU Systems
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